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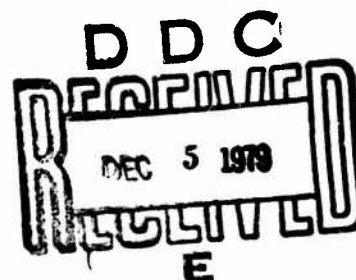
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**PRIMARY ADHESIVELY BONDED STRUCTURE TECHNOLOGY
(PABST)**

General Material Property Data

*DOUGLAS AIRCRAFT COMPANY
McDONNELL DOUGLAS CORPORATION
LONG BEACH, CALIFORNIA 90846*



SEPTEMBER 1978

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Final Report February 1975 - September 1978

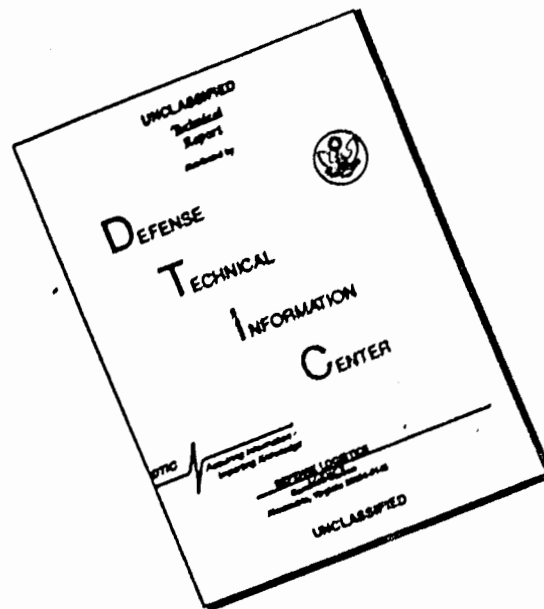
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21. ABSTRACT (Continue on reverse side if necessary and identify by block number) The objective of the program was to investigate state-of-the-art adhesives, adhesive primers, surface treatments, and coatings to determine, through durability testing, a system that could be used to adhesively bond a primary aircraft structure. Test methods were also evaluated during the program. Previous work by Government and industry indicated cyclic-stressed testing in hostile environ- ments and, specifically, high moisture environments reduced the life and		

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durability of a given adhesive system over the same system load to the same level at constant stress.

This program included stressed and cyclic-stressed durability tests for surface treatments, adhesives, and adhesive primers. Coatings were investigated for corrosion protection for the basic alloys used in the program. All durability testing of adhesive bond joint was performed without protective coatings.

The test program compared three types of surface treatments, four corrosion-inhibiting adhesive primers, and four 250°F cure modified epoxy adhesives.

The types of tests included shear, peel, wedge crack, double cantilever beam, creep, shear modulus, and double lap shear. Tests were performed after exposure to fluids and high humidity. Cyclic-stressed testing was performed on modified lap shear specimens in a high humidity environment. Both stressed and unstressed specimens were exposed at a beach site.

Conclusions from this test program include:

- (a) Stressed durability testing is required to determine if a given surface treatment is durable,
- (b) Normal industry accepted processing tolerances on surface treatments are too wide to produce consistently durable bonded joints;
- (c) The handling of surface treated details must be designed to prevent mechanical damage and contamination.
- (d) Phosphoric acid anodize process was the surface treatment most tolerant to processing variations. It was, in addition, inspectable; which is an advantage over the Forest Products Lab (FPL) etch.
- (e) Adhesive primers from the different suppliers demonstrated a large variation in environment resistance and corrosion protection capabilities.
- (f) The type of carrier used in the adhesive films has a large effect on the moisture resistance of the adhesives.
- (g) Cyclic stressed testing in a hostile environment is necessary to determine the optimum adhesive durability.
- (h) Slow cycle testing of specimens configured to typical aircraft type structural design verifies the ranking of the adhesive systems in the laboratory tests.

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PREFACE

This report contains a compilation of test results obtained during the surface treatment tests and adhesive selection tests for the Primary Adhesively Bonded Structure Technology (PABST) program.

The information contained in this report was obtained during the period of February 1975 to September 1978.

This work was performed under Air Force Contract F33615-75-C-3016, and administered by AFFDL. J. Florence is the Project Monitor.

The work described in this report was directed by E. W. Thrall, Program Manager, PABST, Douglas Aircraft Co.

This report was compiled by R. W. Shannon, PABST Project Manager for Materials and Process Engineering, Douglas Aircraft Co.

Contributors included P. Stifel and R. Beger of McDonnell Aircraft Co., for technical assistance and testing of the original cycle stressed environmentally exposed specimens; Drs. E. J. Hughes and J. L. Rutherford of The Singer Co., for shear modulus on the adhesives; Dr. R. J. Sunderland of Douglas Aircraft, for Auger analysis of the treated aluminum surfaces and moisture permeability tests; Dr. J. Carpenter of McDonnell Aircraft Co., for the chemical analysis of the adhesive.

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SECTION I

INTRODUCTION

The use of adhesive bonding in components of aircraft structure has increased dramatically over the last 15 years to the point where most aircraft delivered today utilize some degree of adhesive bonding. However, these applications have been confined primarily to secondary structure where the adhesive bond stress is a low percentage of the adhesive shear strength. This experience with secondary structure has led to the recognition that problems with adhesive bond durability, inspection, and the effects of defects must be solved before adhesive bonding can be extensively used on primary structure.

Extensive government and industry exploratory programs over the past few years have resulted in improved adhesives, primers, and surface preparation as well as improved laboratory test techniques that can closely simulate the type and nature of use in service. In addition, nondestructive inspection techniques for adhesive bonds have been vastly improved. These developments have provided confidence that a final validation program should be pursued to prove the adequacy of adhesive bonding for primary structure.

A series of interrelated Air Force-sponsored programs has been designed to obtain additional bond durability data on coupons and components, provide data on sonic fatigue resistance of bonded structure, develop the necessary manufacturing, field, and depot repair methods, and verify bondline defects.

In February of 1975, Douglas Aircraft Company, under contract to the Air Force, initiated a technology validation program for primary adhesive bonded structures (PABST). This program was conducted (1) to perform a preliminary design, (2) to perform detail design, (3) to fabricate test articles, and (4) to perform coupon, component, and full-scale fatigue, static, and damage-tolerance testing. The baseline configuration was a 42-foot section of the fuselage of the YC-15 airplane. The objective of PABST was to validate that application of adhesive bonding could result in substantial cost and weight savings when compared to conventional fabrication techniques, while providing significant improvements in structural safety and durability.

This report covers the effort of Materials & Process Engineering in the testing of surface treatments coatings and adhesives verifying their uniformity, processability, and environment resistance. All test data in this report were generated on non-Alclad aluminum alloys unless otherwise stated.

The first major effort of the program was to investigate two types of preparing aluminum surface treatments that are widely accepted in industry as being superior and more consistent than the standard Forest Products Laboratory etch. The phosphoric acid anodize developed by the Boeing Commercial Aircraft Co. had been tested by the Air Force under contracts with Boeing and had produced consistent environment resistance bonding surfaces. The chromic acid anodize of the Bell Helicopter Co. and the chromic acid anodize of the McDonnell Aircraft Co. were also considered excellent surface preparations for adhesive bonding of aluminum. These three systems were the primary systems investigated in this program. It is known that there are many other variations of both anodize and etches that are used with success in the industry, but not all systems could be considered for an indepth investigation on this program for obvious reasons. We considered these three systems as being well known "state of the art" in the industry and viable candidates for producing consistent environment resistant surfaces.

The adhesives for this investigation were selected for similar reasons. The base line structure used in the PABST program for demonstration was a typical fuselage section of a cargo type aircraft. The maximum temperature exposure to be expected in normal operation should not exceed 180°F (82°C) nor be exposed to temperatures below -70°F (-57°C). Normal operation could include periods of hot and wet conditions as well as cool and dry. Several adhesive systems developed during the late 1960's and early 1970's were considered to be well designed to be durable in these types of environments. Each adhesive system is used in conjunction with a corrosion inhibiting adhesive primer which can be expected to help optimize the corrosion resistance of the aluminum adherends used in the structure. The four adhesive systems selected for investigation on this program were of this type and are all 250°F (121°C) cure systems. As with the surface treatments, there are many excellent adhesive systems used in industry that have, historically and in test, demonstrated good properties and environment resistance. Again, the

number of systems that could be tested in depth had to be limited to keep the scope of the program manageable. Since the numbers of surface treatments and adhesives used in this program was limited and the testing was directed toward a specific end product and its operating parameters, the information contained in this report should be considered as such and should be used only to assist in deciding what types of tests are required or need to be developed to satisfy the requirements of the project under consideration.

It will become apparent to the reader that several types of tests are required to show some of the anomalies that can exist in these complicated processing and material systems.

The basic criteria for adhesive and surface treatment selection formed around those systems that indicated the most resistance to tests that are stress and moisture oriented. Tests like the double cantilever beam, and lap shear cycle stressed in 140°F and 100% relative humidity carried more weight for system selection than simple immersion tests and static ultimate tests. The end article of the PABST Program is exposed to cyclic loading from a pressurized fuselage and hot moist environment especially in the bilge area.

The data in this report is a compilation of the material tests conducted in the PABST Program by Douglas Aircraft Co. and its subcontractors. Other organizations, including the adhesive manufacturers, have been encouraged to also test and compare their test results with those developed by Douglas.

SECTION II

SURFACE TREATMENT STUDIES

2. Introduction

At the beginning of the program, the first effort was to select the surface treatment that would be used for all subsequent testing of the adhesives. The criteria for the selection of the surface treatment was that the system must be "state of the art". Not all variations of "state-of-the-art" systems could be investigated due to the time schedule that had to be met for starting the adhesive tests. The systems selected for test were well known in the United States and the basic properties were promising based on other test programs by the Air Force and industry.

2.1 Process Selection

Three surface treatments for adhesive bonding on the program were selected for study. They were systems that historically had been used with success in industry or were new developments in industry and that had, by test, indicated a high level of resistance to the environmental conditions the primary structure of the PABST program was expected to be exposed to. These processes included two different procedures for chromic acid anodizing and a procedure for phosphoric acid anodizing. They were as follows:

- a. Phosphoric Acid Anodize, Spec. BAC5555, Boeing Co.
- b. Chromic Acid Anodize, Spec. BPS FW4352, Rev. A, Bell Helicopter Co.
- c. Chromic Acid Anodize, Spec. PS13201, McDonnell Douglas, Inc.

The sulfuric acid-sodium dichromate etch, commonly called Forest Products Lab (FPL) etch, and used widely in industry as a standard, was considered in the program only as a baseline control treatment. The specification used for the preparation of this etch was the Boeing Spec. BAC5514. Some tests were made to compare a fresh solution with the BAC5514 specification solution to "high-light" some of the problems that can be encountered when improper or inadequate process control procedures are used. All testing in this portion of the program was done on bare alloys.

2.2 Adhesion Evaluation Test Methods

The primary evaluation criteria for bond durability of the surface treatments was the wedge crack propagation test. This type of specimen is used by the Boeing Co. as an in-process control test. The advantages are that it is a stressed test in an environment which will indicate problems with a given surface treatment and its environmental resistance characteristics. These characteristics cannot be seen on static type tests such as lap shear specimens. Tests of other known types of specimens would take a longer exposure time for the environment to effect the bond joint. The test specimen configuration is shown in Figure 1. The specimen is fabricated with two plates of the selected alloys which is preferred to be that of the alloy that the specimen is being treated with and is to represent. The plates are processed through the surface treatment with the panels it is to represent and connected in series electrically in the tank to guarantee exposure to the same current as the panel and details it represents. The panels used in the laboratory tests are processed to the parameters under test at the time. These panels, after surface treatment, are primed with the representative primer under test. The primer application procedure will be covered in other sections of this report. The adhesive system used for the surface treatment studies was FM73 mat unless otherwise specified with the BR127 adhesive primer. The vendor (American Cyanamid Co.) standard primer application and bonding procedures were adhered to during the surface treatment tests. Investigations into the primer application tolerances and adhesive bonding tolerances are covered elsewhere in this report.

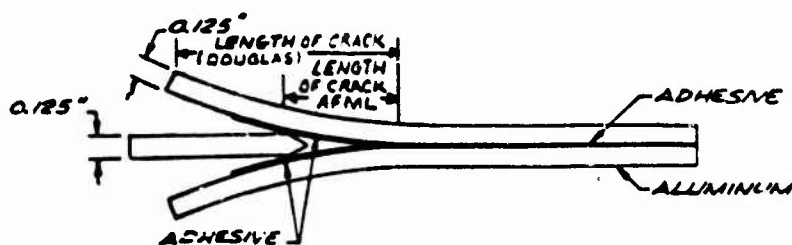


FIGURE 1. WEDGE CRACK SPECIMEN

2.2 (Cont'd)

After primer application, dry, and adhesive application and cure, the panels were cut into five (5) one inch wide wedge crack test specimens. The wedge was driven into one end of the specimen, and after a stabilizing period of one hour, the length of the crack in the specimens were measured. The specimens were then exposed to 95 - 100% relative humidity at 140°F (temperature and humidity studies are described elsewhere). The crack growth was measured after one (1) hour exposure and then remeasured again after three (3) hours exposure.

The mode of failure exhibited by the specimen has now become the major consideration when examining the wedge crack specimens after environmental exposure. The wedge crack test is basically a surface treatment test. It is a quick indication of adhesion in a moist, warm atmosphere. Since each adhesive system may vary in peel strength and creep resistance, numerical comparisons of crack growth may be misleading. The wedge crack test, also, is not the only test that must be performed to optimize the overall durability of a surface treatment, but can be used for process control on a previously optimized system.

2.3 Chemical Analysis & Solution Control

2.3.1 Alkaline Cleaner

The phosphoric acid anodizing process per BAC5555, one of the surface treatments tested in the program, required the use of an alkaline cleaner as specified in specification BAC5749. BAC5749 is not clear as to which of the many alkaline cleaners listed should be used. For standardization purposes, Turco's 4215S (which is listed in the Boeing document) a non-silicated alkaline cleaner, and which is presently used by the Douglas production metal bonding process, was selected for all surface treatment processes evaluated in this Program. Reducing the number of variables in the surface treating procedures was an important part of the program.

2.3.1 (Cont'd)

Turco's 4215S alkaline cleaner is a proprietary compound and very little information is available about the active ingredients. In order to establish control limits of purchased materials, it was important to know what active ingredients are used to make the Turco 4215S compound.

A request for a list of the active ingredients in Turco 4215S cleaner was submitted to the Turco Division of Purex. The list of active ingredients in Turco 4215S alkaline cleaner as submitted by Purex is on file in the Douglas Aircraft Co. laboratory.

The laboratory solution make-up, chemical analysis for process control, date of analysis, and necessary additions including dates are shown in Appendix A.

2.3.2 Deoxidizing Solutions

At the time of the tests, there were two deoxidizers allowed by the Boeing BAC5555 specification for use in preparing parts for phosphoric acid anodizing. These were the FPL etch, a sulfuric acid-sodium dichromate solution, and Amchem #6-16 deoxidizer. The Amchem deoxidizer was chosen for use on the PABST Program instead of the FPL etch for the following reasons:

- a. It can be used to remove heavy oxide deposits.
- b. It can be used in existing laboratory and production tanks without installation of new tank liners.
- c. It is operated at ambient temperature.

A procedure was set up like that used for the alkaline cleaner in an effort to limit and reduce the variables in the test program. To permit maintaining the solution at its optimum during the test programs, a request was made to Amchem Products, Inc., Ambler, Pennsylvania for the active ingredients in the #6 makeup solution and the #16 replenisher solution. The list of active ingredients in the #6 and the #16 deoxidizer as submitted by Amchem Products is on file in the DAC laboratory.

2.3.2 (Cont'd)

A Douglas developed deoxidizer #1051 was used for all specimens processed with chromic acid anodize per McDonnell Douglas P.S. 13201.

A sulfuric acid-sodium dichromate solution at 140°-160°F was used to deoxidize all specimens processed with the Bell helicopter chromic acid anodize per BPS FW4352, Rev. G.

The laboratory solution makeup, chemical analysis for process control, date of analysis, and necessary additions, including dates are shown in Appendix A for all the deoxidizers used in the test program.

2.4 Temperature and Humidity Study

Several companies have used different temperatures when conducting environment exposure tests on wedge crack, stressed lap shear and cycle stressed lap shear specimens. Douglas performed wedge crack tests at four different temperatures and at 100% RH to determine which temperature should be used as a standard for the wedge crack specimens in this program. Besides the results of these tests, the environment to be expected in the end article was considered. This environment would include high temperatures on the ground in a moist (wet bilge) condition with stress on the bonded joint (landing gear, wing, and cargo loads).

To establish the optimum temperature to conduct the wedge crack propagation test, two sets of eight (8) 6 x 6 x 0.125 inch 2024-T3 and 7075-T6 panels were alkaline cleaned, deoxidized and phosphoric acid anodized. The concentration of the anodize solution was maintained in the middle of the concentration range as shown in Table 1.

TABLE 1
PHOSPHORIC ACID ANODIZE PARAMETERS

VARIABLES	PROCESSING LIMITS		
	LOWER	MEDIAN	UPPER
Acid Concentration(oz/gal)	11	13.5	16
Voltage (DC)	8	10	12
Solution Temp °F	65	77.5	90
Anodizing Time (minutes)	20	22.5	25

2.4 (Cont'd)

The detailed procedure for phosphoric acid anodizing the 2024-T3 and 7075-T6 panels, including dates, actual concentration, temperature, time and voltages for all processes are recorded in Appendix B.

The anodized panels were bonded using the FM73/BR127 adhesive and primer system. The temperature evaluation was conducted in 95-100% relative humidity at temperatures of 100°F, 120°F, 140°F and 160°F.

The detailed bonding procedure and humidity test procedure are recorded in Appendix C.

The wedge crack propagation test at the four temperatures for the FPL, etched panels and the phosphoric acid anodized panels are shown in Tables 2, 3, 4 and 5.

2.4 (Cont'd)

TABLE 2
TEMPERATURE AND HUMIDITY TEST RESULTS

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH IN INCHES	AFTER 1 HR. AT TEMP. ADDED GROWTH	AFTER 3 HR. AT TEMP. ADDED GROWTH	COMMENTS
7FPL1-1-1	3-13-75	.0055"	100	2.20	.400"		
-2		.0060"		2.17	1.025	Test Discontinued	
-3		.0058"		2.19	1.090		
-4		.0056"		2.21	.825		
-5		.0037"		2.24	.290		Adhesive Failure
7FPL1-2-1		.0030"	120	2.13	.030		
-2		.0048"		2.17	.360	Test Discontinued	
-3		.0050"		2.14	.490		
-4		.0041"		2.18	.162		
-5		.0020"		2.23	.060		
7FPL1-3-1		.0040"	140	2.23	.060	.075"	
-2		.0065"		2.23	.077	N.C.	
-3		.0070"		2.24	.090	.117	
-4		.0068"		2.14	.077	.139	
-5		.0055"		2.22	.095	.110	
7FPL1-4-1		.0040"	160	2.24	.119	Test Discontinued	
-2		.0060"		2.22	.118		
-3		.0060"		2.24	.112		
-4		.0060"		2.13	.167		
-5		.0045"		2.13	.195		

Relative Humidity, 95-100%
7075-T6
FPL ETCH

N.C. = No Change

2.4 (Cont'd)

TABLE 3
TEMPERATURE AND HUMIDITY TEST RESULTS

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH IN INCHES	AFTER 1 HR. AT TEMP. ADDED GROWTH	AFTER 3 HR. AT TEMP. ADDED GROWTH	COMMENTS
2FPL1-1-1	3-13-75	.0050"	100	2.22	1.965"	Test Discontinued	↑
-2		.0060"		2.22	2.735		
-3		.0070"		2.21	2.175		
-4		.0060"		2.13	1.935		
-5		.0040"		2.23	.260		
2FPL1-2-1		.0060"	120	2.17	.175		Adhesive Failure
-2		.0060"		2.14	.280		
-3		.0068"		2.11	.740		
-4		.0060"		2.10	.560		
-5		.0060"		2.18	.079		
2FPL1-3-1		.0055"	140	2.11	.810		
-2		.0070"		2.17	1.025		
-3		.0050"		2.10	.828		
-4		.0071"		2.23	.700		
-5		.0055"		2.37	.140		
2FPL1-4-1		.0055"	160	2.13	.600	↓	
-2		.0060"		2.08	.695		
-3		.0060"		2.14	.730		
-4		.0052"		2.20	.155		
-5		.0042"		2.19	.635		

Relative Humidity, 95-100%
2024-T3
FPL Etch

2.4 (Cont'd)

TABLE 4
TEMPERATURE AND HUMIDITY TEST RESULTS

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH IN INCHES	AFTER 1 HR. AT TEMP. ADDED GROWTH	AFTER 3 HOURS AT TEMP. ADDED GROWTH	EXTENDED EXPOSURE ADDED GROWTH	COMMENTS
7PIR-A-100-1	3-12-75	.0050"	100	2.18	.000"	N.C.	68 Hr.	Slight porosity not necessarily crack extension (after 3 Hr.)
-2		.0040"		2.15	.030	N.C.	N.C.	
-3		.0040"		2.14	.050	.070"	N.C.	
-4		.0048"		2.15	.045	N.C.	N.C.	
-5		.0050"		2.16	.040	.047	N.C.	
7PIR-B-120-1	3-13-75	.0060"	120	2.12	.015	.047	18 Hr.	Cohesive Failure
-2		.0072"		2.14	.027	N.C.	N.C.	
-3		.0068"		2.12	.035	N.C.	N.C.	
-4		.0057"		2.10	.070	N.C.	N.C.	
-5		.0080"		2.16	.020	N.C.	.078"	
7PIR-C-140-1	3-12-75	.0055"	140	2.20	.040	N.C.	68 Hr.	
-2		.0050"		2.30	.028	N.C.	N.C.	
-3		.0060"		2.20	.038	N.C.	.055"	
-4		.0072"		2.20	.050	N.C.	.054	
-5		.0065"		2.18	.038	N.C.	.089	
7PIR-D-160-1	3-13-75	.0055"	160	2.19	.068	N.C.	68 Hr.	
-2		.0045"		2.21	.070	.095	N.C.	
-3		.0065"		2.17	.046	N.C.	N.C.	
-4		.0065"		2.18	.015	N.C.	.070	
-5		.0060"		2.18	.060	N.C.	.130	

N.C. = No Change

Relative Humidity, 95-100%
Phosphoric Acid Anodize
7075-T6

TABLE 5
TEMPERATURE AND HUMIDITY TEST RESULTS

SPECIFIC I.D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH IN INCHES	AFTER 1 HR. AT TEMP. ADDED GROWTH	AFTER 3 HOURS AT TEMP. ADDED GROWTH	EXTENDED EXPOSURE ADDED GROWTH	COMMENTS
2PIR-A-100-1 -2 -3 -4 -5	3-12-75 ↓	.0040"	100 ↓	2.15	.008"	.043"	N.C.	Cohesive Failure ↓
		.0035"		2.15	.008	.025	N.C.	
		.0035"		2.14	.020	.082	N.C.	
		.0040"		2.15	.045	N.C.	N.C.	
		.0038"		2.15	.028	N.C.	N.C.	
2PIR-B-120-1 -2 -3 -4 -5	3-13-75 ↓	.0023"	120 ↓	2.10	.046	N.C.	18 Hr. N.C.	
		.0036"		2.12	.020	N.C.	N.C.	
		.0037"		2.14	.011	N.C.	N.C.	
		.0032"		2.14	-0-	N.C.	N.C.	
		.0030"		2.12	.040	N.C.	.057"	
2PIR-C-140-1 -2 -3 -4 -5	3-12-75 ↓	.0030"	140 ↓	2.18	.035	N.C.	68 Hr. N.C.	
		.0040"		2.15	.047	N.C.	.074"	
		.0030"		2.20	.050	N.C.	.079	
		.0035"		2.20	.060	N.C.	.082	
		.0045"		2.18	.030	N.C.	.118	
2PIR-D-160-1 -2 -3 -4 -5	3-13-75 ↓	.0038"	160 ↓	2.10	.035	.065	N.C.	
		.0040"		2.12	.075	N.C.	N.C.	
		.0048"		2.10	.060	N.C.	N.C.	
		.0032"		2.13	.055	.095	N.C.	
		.0045"		2.13	.052	N.C.	N.C.	

Relative Humidity 95-100%
Phosphoric Acid Anodize
2024-T3

N.C. = No Change

2.4 (Cont'd)

The test results for the FPL etched specimens were very erratic as can be seen in Tables 2 and 3. However, the 140°F seemed to be adequate for the phosphoric acid anodized specimens. The 140°F temperature at 95-100% relative humidity was chosen to be the test condition for all future wedge crack tests.

2.5 Phosphoric Acid Anodizing Process Parameters Confirmation

The processing parameters for the phosphoric acid anodizing process were confirmed by the use of the factorially designed matrix as shown in Figure 2.

Phosphoric Acid Anodize

		C ₁		C ₂	
		V ₁	V ₂	V ₁	V ₂
T ₁	t ₁	X	λ	X	X
	t ₂	X	X	X	λ
T ₂	t ₁	X	λ	X	X
	t ₂	X	X	X	X

FIGURE 2. TEST MATRIX

In accordance with the test matrix of Figure 2, the effects and the interactions of such anodizing variables as solution concentration (C₁C₂), applied voltage (V₁V₂), solution temperature (T₁T₂), and the anodizing time (t₁t₂) were evaluated. Five (5) wedge crack test specimens for each of the two alloys (2024-T3 and 7075-T6) were prepared for each of the test grids indicated in the matrix diagram. Sixteen data points or 80 wedge crack test specimens per data point were tested in the matrix per alloy.

2.5 (Cont'd)

The variables investigated were the highs and lows of the anodizing parameters shown in Table 1.

The detailed racking, cleaning, deoxidizing procedures, plus the exact temperature, time, voltages and other data that were recorded while processing each condition are reported in Appendix D.

After anodizing, the 6- x 6-inch panels were primed with BR127 adhesive. FM73 adhesive film was applied and the test panels were bonded in an autoclave at 250°F for 90 minutes under 40 psi pressure. All test panel assemblies were nondestructive C-scan inspected. No voids were noted in the bonded test panel assemblies.

The test panel assemblies were then cut into five (5) one-inch-wide test specimens, the wedge driven into one end of the specimen, and after a stabilizing period, of one hour, the opening of the glue line produced by the wedge was measured. The specimens were then exposed to 100% RH at 140°F. The crack growth was measured after one (1) hour and three (3) hours. In addition to the shorter periods some specimens were also exposed for periods of 18 and 168 hours. It appears that the primary crack growth takes place within the first hour and this time was used for data points in the matrix.

The wedge crack growth test results for each condition tested are shown in Appendix D. The same data is shown in the matrix diagram in Figures 3 and 4. Wedge crack tests on the four surface treatments were also exposed to salt spray. After three hours, all the specimens had similar rates of crack growth.

Scanning electron microscope photographs of the surface of pretreated and phosphoric acid anodized specimens is shown in Figures 5 and 6.

2.5 (Cont'd)

		CONC. 11 OZ/GAL		CONC. 16 OZ/GAL	
		VOLTAGE 8	VOLTAGE 12	VOLTAGE 8	VOLTAGE 12
TEMP.	65°F	Time 20 Min.	.000	.107	.036
			.047	.053	.030
			.032	.034	.020
			.013	.048	.042
			.035	.037	.035
	65°F	Time 25 Min.	.025	.053	.060
			.025	.010	.020
			.045	.086	.010
			.032	.062	.000
			.008	.030	.015
TEMP.	90°F	Time 20 Min.	.090	.060	.050
			.094	.040	.047
			.042	.082	.022
			.045	.021	.030
			.105	.058	.037
	90°F	Time 25 Min.	.045	.080	.020
			.035	.094	.008
			.015	.096	.046
			.035	.052	.025
			.040	.063	.006

Numbers indicate inches of crack growth measured after exposure to 140°F and 100% RH for 1 hour

FIGURE 3. PHOSPHORIC ACID ANODIZE MATRIX 2024-T3 BARE WEDGE CRACK

2.5 (Cont'd)

		CONC. 11 OZ/GAL		CONC. 16 OZ/GAL	
		VOLTAGE 8	VOLTAGE 12	VOLTAGE 8	VOLTAGE 12
TEMP.	65°F	Time 20 Min.	.045	.033	.055
			.050	.015	.050
			.065	.020	.050
			.030	.043	.045
			.065	.065	.057
	25 Min.	Time	.053	.088	.017
			.026	.076	.020
			.032	.063	.040
			.015	.155	.020
			.015	.047	.008
TEMP.	90°F	Time 20 Min.	.092	.100	.042
			.112	.075	.045
			.037	.132	.041
			.023	.017	.021
			.051	.061	.010
	25 Min.	Time	.078	.063	.045
			.010	.039	.100
			.005	.030	.090
			.016	.027	.047
			.010	.012	.065

Numbers indicate inches of crack growth measured after exposure to 140°F and 100% RH for 1 hour

FIGURE 4. PHOSPHORIC ACID ANODIZE MATRIX 7075-T6 BARE WEDGE CRACK

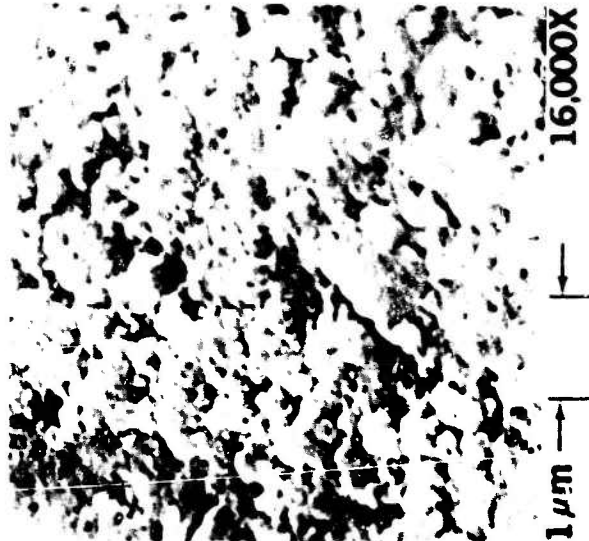
2.6.1 (Cont'd)

TABLE 6 CHROMIC ACID ANODIZE & SEALING PARAMETERS

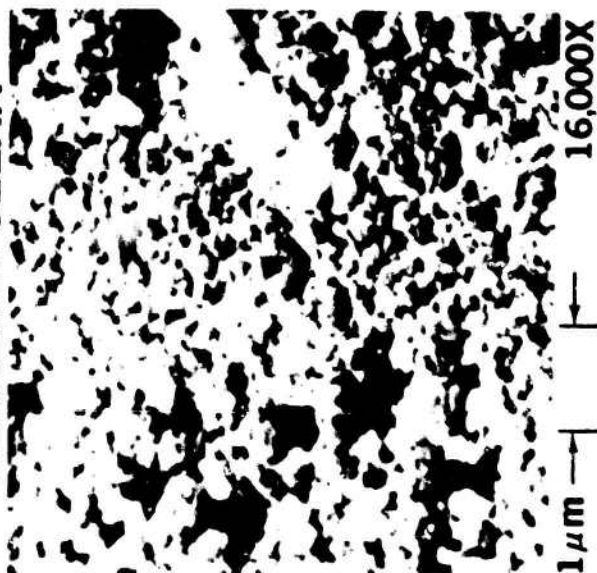
	VARIABLES	PROCESSING LIMITS					
		MDC			BELL HELICOPTER		
		LOWER	MEDIAN	UPPER	LOWER	MEDIAN	UPPER
Anodize	Chromic Acid Conc. (% free acid)	4.5	5.25	6	3	-	-
	Voltage (V)	17	20	23	38	40	42
	Solution Temperature °F	85	95	100	92	93.5	95
	Anodizing Time - Mins	25	35	40	30	32.5	35
Sealing Solution	Chemical	Potassium Dichromate			Chromic Acid		
	MDC-% bw Concentration Bell-ppm	2.5	5	6	75	102.5	120
	Temperature °F	185	195	205	180	182.5	185
	Time - Mins	8	12.5	17	7	8	9
	pH	4.2	5.5	6	2.5	3.15	3.8

One set (2 panels) each of 2024-T3 and 7075-T6 nonclad 6 x 6 x 0.125 inch for the MDC anodizing were processed using the median processing range shown in Table 6 above. The bonded 6- x 6- x .125-inch panels were cut into 5 wedge crack test specimens. The crack growth was measured after one (1) hour and after 3 hours. The above test was repeated for the Bell chromic acid anodizing process. The laboratory solution makeup, chemical analysis for process control, date of analysis, necessary additions, detailed processing sequence, including dates for the MDC and Bell process are shown in Appendix E. The results of the wedge crack tests are shown in Table 7.

SOLVENT CLEAN



ALKALINE CLEAN



DEOXIDIZER

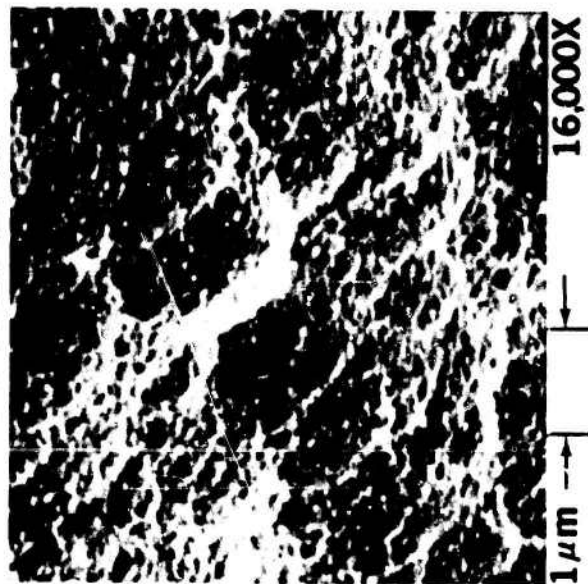


FIGURE 5. PHOSPHORIC ACID ANODIZE PRETREATMENTS

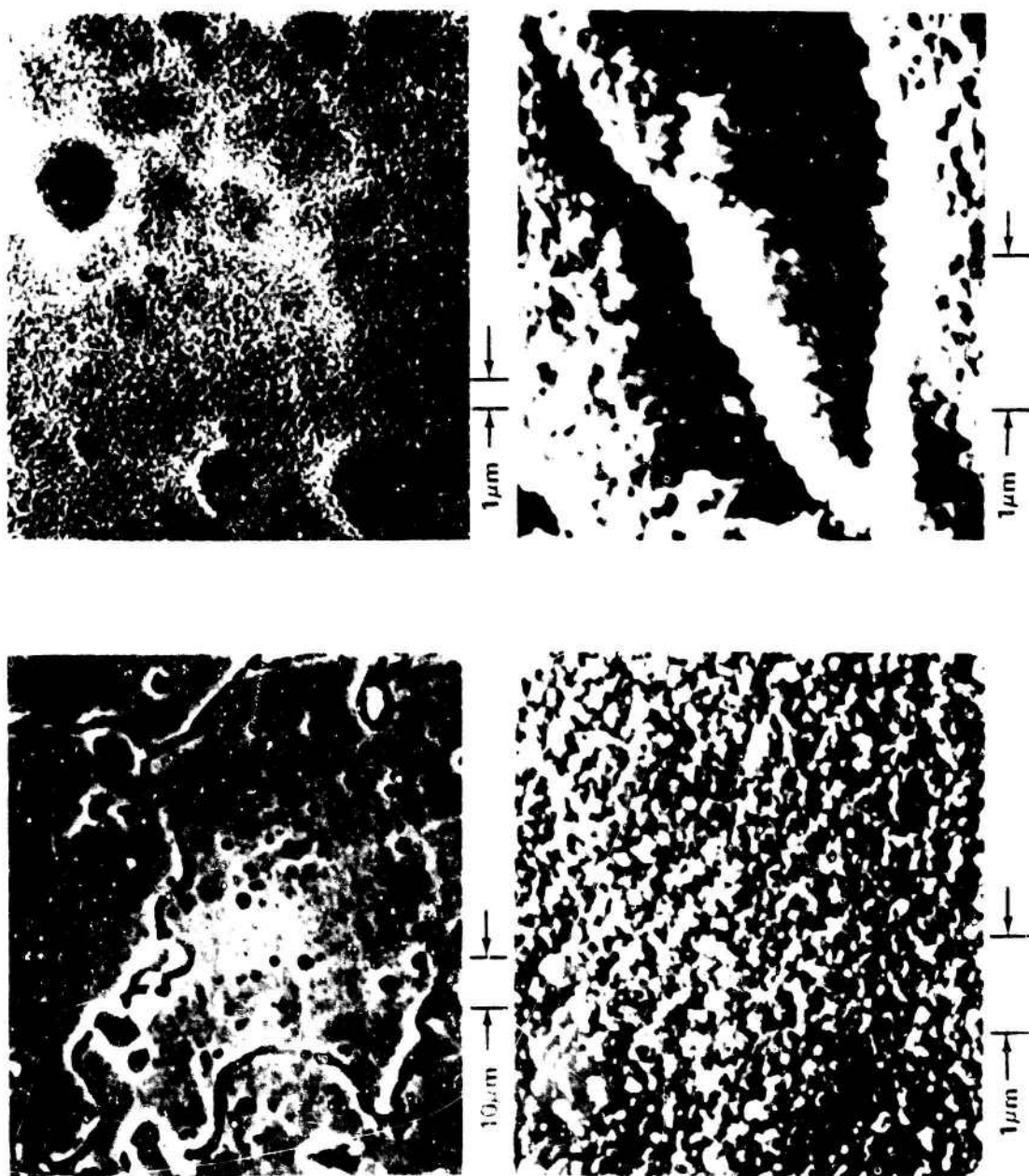


FIGURE 6. PHOSPHORIC ACID ANODIZE

2.6 CHROMIC ACID ANODIZE

Chromic acid anodize was the second type of surface treatment to be tested in the program. A wide usage in industry here and in Europe and generally good service experience made it a viable consideration. It will be shown that the chromic acid anodize by itself has much better corrosion resistance than the phosphoric acid anodize. The question was, could it also produce environment resistant bonded joints to the level necessary for primary structure.

Two systems were considered in this program.

2.6.1 Chromic Acid Anodize Trade Off Study - MDC Versus Bell

A comparison of two chromic acid anodizing processes (McDonnell Douglas per P.S. 13201 and Bell Helicopter Co. per BPS FW4352, Rev. G.) was conducted using their respective media processing range. Both the Bell and MDC chromic acid anodizing and sealing parameters are shown in Table 6. This test was made to decide which system would be used for further evaluation.

It should be noted that the sealing procedure for the chromic acid anodize is a primary consideration in the anodizing acceptability for adhesive bonding. Many investigators believe that an unsealed or low temperature sealed anodize produces the highest strength bonded joint. Others consider the unsealed anodize to be low in corrosion resistance. An indepth investigation into all the parameters of the chromic acid anodize processing to develop an optimized system for primary adhesive bonding is desirable but could not be considered within the scope of this program. We, therefore, tested the two state-of-the-art systems that we felt were representative of chromic acid anodizing for adhesive bonding as used in the United States.

2.6.1 (Cont'd)

TABLE 7

CRACK EXTENSION TEST DATA

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH IN INCHES	AFTER 1 HR. AT TEMP. ADDED GROWTH	AFTER 3 HR. AT TEMP. ADDED GROWTH	COMMENTS
2B4 Bell Chromic (2024)	3-20-75	.0050"	140	2.20	.337"	.390"	Adhesive Failure ↓
		.0059"		2.15	.500	.600	
		.0070"		2.20	.524	.525	
		.0055"		2.21	.690	N.C.	
		.0039"		2.23	.083	.125	
7B4 (7075) Bell		.0048"		2.23	.060	.127	Cohesive Failure ↓
		.0070"		2.22	.077	N.C.	
		.0075"		2.20	.087	N.C.	
		.0072"		2.19	.087	.103	
		.0056"		2.31	.062	N.C.	
2M4 (2024) MDC Chromic		.0039"		2.22	.063	N.C.	
		.0050"		2.18	.062	N.C.	
		.0058"		2.20	.067	N.C.	
		.0058"		2.24	.086	N.C.	
		.0044"		2.14	.160	.183	
7M4 (7075) MDC		.0058"	↓	2.22	.103	N.C.	↓
		.0070"		2.19	.147	N.C.	
		.0080"		2.20	.107	.127	
		.0070"		2.20	.088	N.C.	
		.0059"		2.23	.067	.078	

N.C. = No Change

2.6.1 (Cont'd)

The Bell anodize wedge crack test on the 2024-T3 specimens showed considerable adhesive failures associated with large crack growths. The Bell system on 7075-T6 and the MDC anodize on both 2024-T3 and 7075-T6 did not show any adhesive failure. Because of the adhesive failures of the Bell anodized on 2024-T3 specimen, the MDC chromic acid anodize was chosen for further evaluation.

2.6.2 Chromic Acid Anodize - Sealing Parameters Confirmation

The processing range for the sealing operation of the MDC chromic acid anodizing process was studied by using the factorially designed matrix as shown in Figure 7.

		C ₁		C ₂	
		W ₁	W ₂	W ₁	W ₂
T ₁	t ₁	10	10	10	10
	t ₂	10	10	10	10
T ₂	t ₁	10	10	10	10
	t ₂	10	10	10	10

C = Trace acid or dichromate con.

W = Water W₁ = Deionized

W₂ = Tap

T = Temperature

t = Time

° 2 alloys

° 5 specimens per alloy

FIGURE 7. CHROMIC ACID ANODIZE SEALING MATRIX

All the anodizing was done at the median range as shown in Table 6. In accordance with the test matrix of Figure 7, the effects and the interactions of such variables as the concentration of potassium dichromate (C₁C₂), type of water (W₁W₂), solution temperature (T₁T₂), and the sealing time (t₁t₂) were evaluated. Wedge crack test specimens of each of the two alloys (2024-T3 and 7075-T6 nonclad) were anodized and sealed for each of the test grids indicated in the matrix diagram.

2.6.2 (Cont'd)

The variables and ranges investigated are as follows:

- W_1 - Used deionized water for makeup
- W_2 - Used tap water for make up
- C_1 - Concentration of potassium dichromate - 2.5% by wt.
- C_2 - Concentration of potassium dichromate - 6% by wt.
- T_1 - Temperature - at 185°F
- T_2 - Temperature - at 205°F
- t_1 - Time - at 8 minutes
- t_2 - Time - at 17 minutes

The detailed, cleaning, deoxidizing, anodizing, rinsing and sealing data are recorded in Appendix F.

The results of the sealing evaluation are shown in Figures 8, 9, 10, 11, 12, 13, 14 and 15.

2.6.2 (Cont'd)

		DEIONIZED WATER	
		Conc. 2.5% Pot. Dichromate pH 4.2	Conc. 6.0% Pot. Dichromate pH 6.0
Temp 185°F	Time 8 Min.	.087 AE .057 C .057 C .062 AE .037 C	.536 A .185 A .077 AE .243 A .107 AE
	Time 17 Min.	.070 AE .052 C .088 AE .097 AE .125 AE	.087 AE .790 A .735 A .782 A 1.267 A
Temp 205°F	Time 8 Min.	.072 C .072 C .107 C .057 C .067 C	.868 A .927 A 1.043 A 1.111 A .680 A
	Time 17 Min.	.102 AE .060 AE .077 AE .070 AE .097 AE	.129 AE .101 AE .093 AE .121 AE .115 AE

7075-T6 Bare
Wedge Crack
Failure Mode:

Added crack growth in inches
Exposure = 140°F and 100% RH
for 1 hour
A = Adhesive
C = Cohesive
AE = Adhesive Failure
Edge Only

FIGURE 8. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

		DEIONIZED WATER	
		Conc. 2.5% Pot. Dichromate pH 4.2	Conc. 6.0% Pot. Dichromate pH 6.0
Temp 185°F	Time 8 Min.	.115 AE .143 AE .065 AE .097 AE .075 AE	.068 AE .068 AE .950 A 1.420 A .830 A
	Time 17 Min.	.084 AE .095 AE .056 AE .107 AE	.075 AE .092 AE .085 AE .102 AE
Temp 215°F	Time 8 Min.	.080 AE .057 C .065 C .073 C .077 C	.225 A .854 A .845 A .072 AE .092 AE
	Time 17 Min.	.072 AE .062 AE .085 AE .087 AE .087 AE	.094 AE .097 AE .117 AE .128 AE .135 AE

2024-T3 Bare

Wedge Crack

Failure Mode:

Added crack growth in inches

Exposure = 140°F and 100% RH
for 1 hour

A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 9. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

TAP WATER			
		Conc. 2.5% Pot. Dichromate pH 5.8	Conc. 6.0% Pot. Dichromate pH 5.8
Temp 185°F	Time 8 Min.	.103 AE .100 AE .093 AE .113 AE .067 AE	.070 AE .050 C .058 AE .075 AE ★
	Time 17 Min.	.092 AE .092 AE .102 AE .115 AE .111 AE	1.125 A 1.450 A 2.100 A .070 AE 1.300 A
Temp 205°F	Time 8 Min.	.085 AE .103 AE .105 AE .103 AE .077 AE	1.570 A 1.897 A 1.362 A 1.304 A .849 A
	Time 17 Min.	.165 AE .085 AE .117 AE .088 AE .105 AE	1.572 A 1.465 A 1.708 A 1.825 A 1.400 A

7075-T6 Bare Added crack growth in inches

Wedge Crack Exposure = 140°F and 100 RH
for 1 hour

Failure Mode: A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 10. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

		TAP WATER	
		Conc. 2.5% Pot. Dichromate pH 5.8	Conc. 6.0% Pot. Dichromate pH 5.8
Temp 185°F	Time 8 Min.	.020 C .063 C .075 C .130 AE .090 AE	.025 AE .012 C .045 C .055 C .032 C
	Time 17 Min.	.075 AE .091 AE .113 AE .057 AE .080 AE	1.140 A .020 AE 1.250 A 1.850 A .087 AE
Temp 205°F	Time 8 Min.	.050 AE .800 A 1.120 A 1.110 A .635A	2.129 A 1.792 A 2.131 A 2.340 A 2.611 A
	Time 17 Min.	.117 AE .306 A .713 A .079 AE .168 AE	2.402 A 2.258 A 2.454 A 2.255 A 3.654 A

Added crack growth in inches

2024-T3 Bare

Wedge Crack

Failure Mode:

Exposure = 140°F and 100% RH
for 1 hour

A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 11. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (cont'd)

		DEIONIZED WATER	
		Conc. 2.5% Pot. Dichromate pH 4.8	
		7075-T6 Bare	2024-T3 Bare
Temp 205°F	Time 8 Min.	1.630 A 1.613 A 1.676 A 1.682 A 1.639 A	2.454 A 2.345 A 2.605 A 2.048 A 2.550 A
	Time 17 Min.	.800 A 1.340 A 1.355 A .719 A .587 A	2.520 A 1.700 A 1.770 A 2.670 A 1.480 A

Retest using glass containers for seal solution

Added crack growth in inches

Wedge Crack Exposure = 140°F and 100% RH for 1 hour

Failure Mode: A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 12. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

		DEIONIZED WATER		
		Conc. 2.5% Pot. Dichromate pH 3.8		Conc. 6.0 pH 3.9
Temp 205°F	Time 8 Min.	Glass	Fiberglass	
	Time 17 Min.			
		.052 C	.067 C	.062 C
		.067 C	.085 C	.050 C
		.050 C	.050 C	.067 C
		.084 C	.037 C	.033 C
		.069 C	.062 C	.039 C
		.087 C	.066 C	.092 C
		.103 AE	.078 C	.080 C
		.240 AE	.057 C	.046 C
		.060 C	.091 C	.097 C
		.092 C	.087 C	.112 C

Retest: Glass Vs. Fiberglass Sealing Tanks

Added crack growth in inches

7075-T6 Bare

Wedge Crack

Failure Mode:

Exposure = 140°F and 100% RH
for 1 hour

A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 13. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

		DEIONIZED WATER	
		Conc. 2.5% Pot. Dichromate pH 4.0	Conc. 6.0% Pot. Dichromate pH 4.0
Temp 185°F	Time 8 Min.	.049 C .054 C .077 C .053 C .090 C	.078 C .078 C .057 C .110 C .058 C
	Time 17 Min.	.137 C .070 C .062 C .052 C .033 C	.042 C .032 C .067 C .118 C .060 C
Temp 205°F	Time 8 Min.	.070 C .078 C .070 C .060 C .067 C	.057 C .050 C .083 C .095 C .122 C
	Time 17 Min.	.102 C .062 C .103 C .047 C .073 C	.077 C .082 C .130 C .101 C .100 C

7075-T6 Bare

Edge Crack

Failure Mode:

Added crack growth in inches

Exposure = 140°F and 100% RH
for 1 hour

A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 14. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

		DEIONIZED WATER	
		Conc. 2.5% Pot. Dichromate pH 4.0	Conc. 6.0% Pot. Dichromate pH 4.0
Temp 185°F	Time 8 Min.	.075 C .056 C .047 C .040 C .100 C	.052 C .063 C .085 C .046 C .027 C
	Time 17 Min.	.100 C .070 C .038 C .044 C .033 C	.039 C .052 C .038 C .067 C .043 C
Temp 205°F	Time 8 Min.	.057 C .060 C .070 C .067 C .068 C	.050 C .060 C .052 C .052 C .052 C
	Time 17 Min.	.548 A .610 A .050 AE .090 AE .070 AE	.003 C .067 C .037 C .070 C .091 C

2024-T3 Bare

Wedge Crack

Failure Mode:

Added crack growth in inches
 Exposure = 140°F and 100% RH
 for 1 hour

A = Adhesive

C = Cohesive

AE = Adhesive Failure at edge only

FIGURE 15. CHROMIC ACID ANODIZE SEALING MATRIX

2.6.2 (Cont'd)

The test results shown in Figures 8 - 15 includes in addition to crack growth (in inches) the failure mode of each specimen. The wedge crack specimens after being exposed to 95-100% R.H. at 140°F and the crack growth was recorded, the wedge was driven into the specimen until it separated as shown in photos, Figure 16, 17 and 18. The dark grey area seen in Figure 16 is the cured adhesive system. The failure mode in Figure 16 is referred to as cohesive (C). A cohesive failure indicates that the adhesive strength of the adhesive system to the treated aluminum is greater than the cohesive strength of the FM73 adhesive. The cohesive failure is considered to be the ultimate in adhesion as a result of surface treatment. The failure mode in Figure 17 is referred to as adhesive (A). An adhesive failure is considered to be worst type of adhesion as a result of surface treatment.

Specimens having the failure identified as "AE" indicate a minor adhesive failure at the edge of the specimen in the crack zone. Though these failures are minor, they are considered important since this edge area of the specimen are the points where the anodize/adhesive faying surface are immediately exposed to the test environment. It also is an indication of method of specimen preparation and the effects of cutting bond lines in manufacturing. Brittle anodizes that fracture during machining can be sources of included areas for the starting of corrosion.

WEDGE
CRACK
TEST

7075-T6
NONCLAD
ALUMINUM
ALLOY

SALT SPRAY
EXPOSURE
100 HRS
AT 94°F

PHOSPHORIC
ACID
ANODIZE

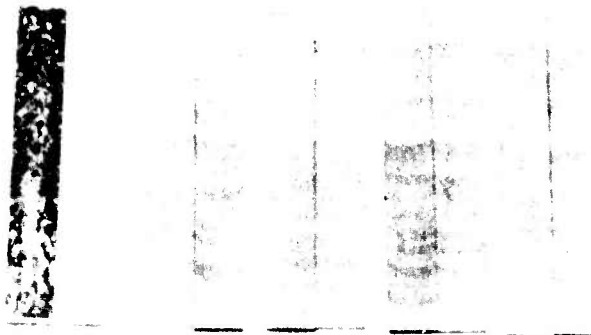


FIGURE 16. PHOSPHORIC ACID ANODIZE - SALT SPRAY EXPOSURE

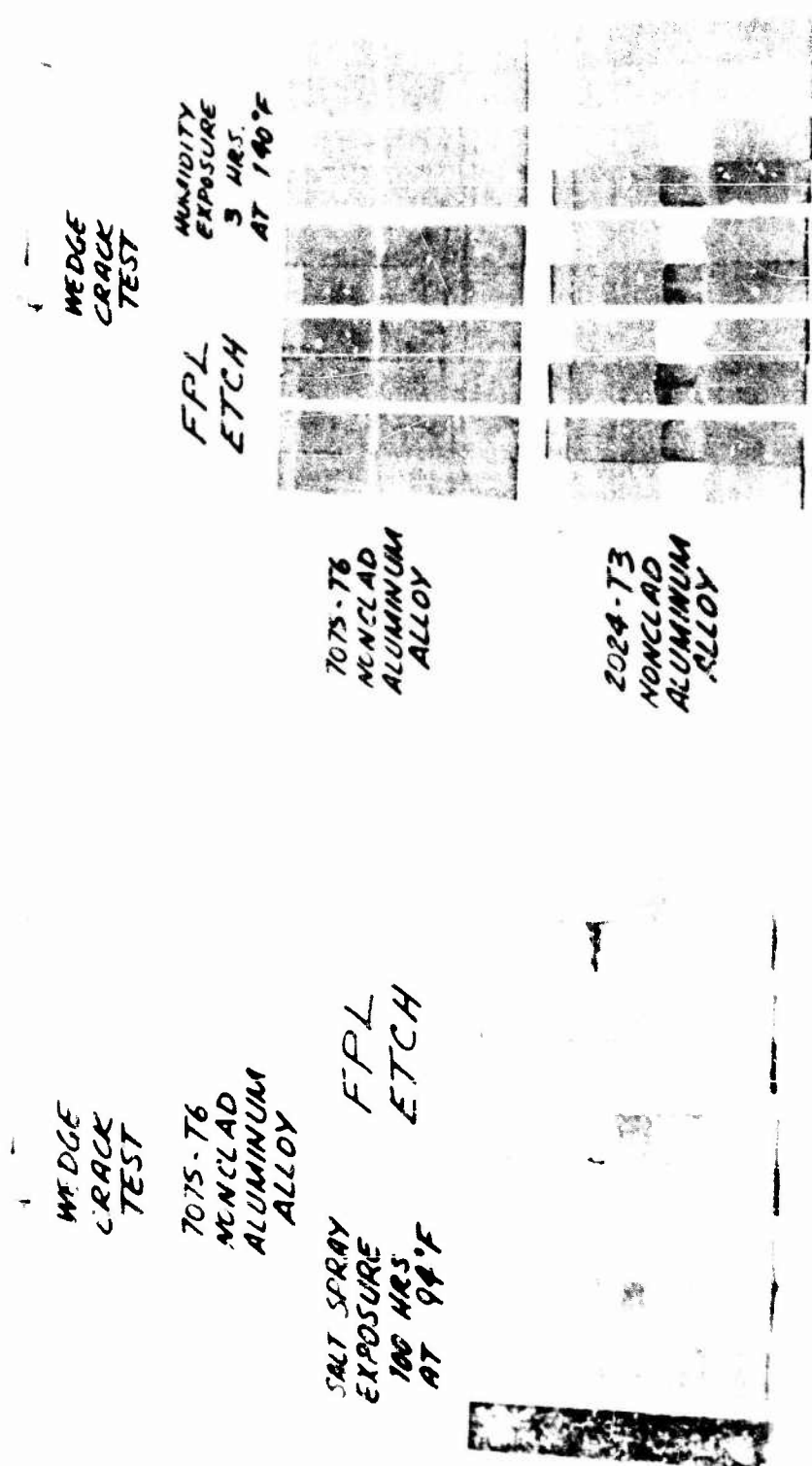


FIGURE 17. FPL ETCH-HUMIDITY EXPOSURE

FIGURE 18. FPL ETCH-HUMIDITY EXPOSURE

2.6.2 (Cont'd)

The data shown in Figure 12 is the result of a retest to determine if the many adhesive failures were due to contamination of the seal solution from fiberglass resin leeching out of the tank walls. Deionized water with 2.5% potassium dichromate, pH 4.8, and sealed at 205°F for 8 and 17 minutes were repeated on both 7075-T6 and 2024-T3 nonclad specimens. The solution was made up in a 6 liter battery jar to eliminate any contamination.

After reviewing the results shown in Figure 12 the test was again repeated using the glass container and the fiberglass container at both concentrations of potassium dichromate (2.5% and 6.0%). The pH of the seal solutions was not adjusted but left as made up. The results were as shown in Figure 13.

As a result of the repeated test the complete sealing matrix was repeated using the deionized water seal solution at a pH of 4.0. All these tests have shown that the pH of the dichromate seal solution is of greater importance than originally believed. The data indicates that poor resistance to moisture is a product of high pH (4.8 and above) and to a lesser degree to concentration of the solution and the temperature.

The optimum processing range for the sealing operation for the MDC chromic acid anodize as confirmed in the evaluation is as shown in Table 8.

TABLE 8
OPTIMUM SEALING CONDITIONS

Potassium Dichromate ($K_2Cr_2O_7$)	- 4.0% to 6.0%
Sealing Time	- 8 to 12 minutes
Temperature	- 185° to 195°F
Water	- Deionized
pH	- 3.8 to 4.2

2.6.3 Chromic Acid Anodizing Process Parameters Confirmation

The processing parameters for the MDC chromic anodizing process were evaluated by the use of the factorially designed matrix as shown in Figure 2 for phosphoric acid anodize.

In accordance with the test matrix of Figure 2, the effects and the interactions of such anodizing variables as solution concentration (C_1C_2), applied voltage (V_1V_2), solution temperature (T_1T_2) and the anodizing time (t_1t_2) were evaluated.

Five (5) wedge crack test specimens for each of the two alloys (2024-T3 and 7075-T6 nonclad) were prepared for each of the test grids indicated in the matrix diagrams. The variables investigated were the highs and lows of the anodizing parameters as shown in Table 9:

TABLE 9
CHROMIC ACID ANODIZE PARAMETERS

VARIABLES	PROCESSING LIMITS		
	LOWER	MEDIAN	UPPER
ACID CONCENTRATION (OZ/GAL)	6.0	7.0	8.0
VOLTAGE (DC)	17	20	23
SOLUTION TEMPERATURE (°F)	85	92.5	100
ANODIZING TIME (MINUTES)	25	35	40

2.6.3 (Cont'd)

Another set of panels (6 x 6 x .125 inch) for each alloy was processed at low concentration, low temperature, low voltage and time. The anodize thickness of the specimens were measured using a Permascope tester type EC. The anodize film thickness of the panels were as shown in Figure 19.

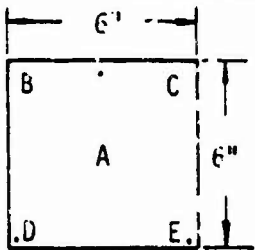
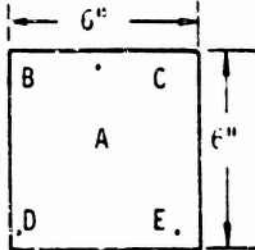
PANEL I.D.	COATING THICKNESS IN INCHES	
Panel #1	A = 0.00001"	
	B = 0.00005"	
	C = 0.00003"	
	D&E = 0.00002"	
Panel #2	A = 0.00001"	
	B&C = 0.00002"	
	D = 0.00001"	
	E = 0.00003"	

FIGURE 19. ANODIZE THICKNESS 7075-T6 ALLOY NONCLAD

The anodize film thickness on the 2024-T3 nonclad alloy could not be measured.

The specimens used to determine anodize film thickness were subjected to salt spray test. The salt spray test is the 5% solution specified in ASTM B 117.

It was decided to conduct some preliminary anodizing tests before beginning the third quadrant of the MOC chromic acid anodizing matrix. The third quadrant of this matrix specifies a high acid concentration at low temperature. To determine if these conditions produced the pink iridescence (poor or thin anodic film), 6 x 6 x 0.125 inch panels of 2024-T3 nonclad and 7075-T6 nonclad were anodized.

2.6.3 (Cont'd)

The concentration of the chromic acid was increased to 8 oz/gal and the temperature was lowered to 85°F and the specimens were anodized at 17 volts for 25 minutes. The specimens were sealed using the median seal range. The appearance of the 2024 alloy after anodizing and sealing was a uniform semi-bright, speckled pattern of beige to greenish beige shade, with some iridescence. The appearance of the 7075 alloy was the same as the 2024 except speckling was more distinct, with some iridescence but a distinct magenta shade. Both panels were subjected to 5% salt spray test.

Because the anodic coating of these tests resulted with enough iridescence to suggest a thin anodic coating, it was decided to raise the temperature of the bath from 85°F to 90°F for the confirmation tests involving the higher acid concentrations.

		CONCENTRATION 6 OZ/GAL FREE ACID		CONCENTRATION 8 OZ/GAL FREE ACID	
		VOLTAGE 17	VOLTAGE 23	VOLTAGE 17	VOLTAGE 23
TEMP 85°F	TIME 25 MINUTES	0.944 A 1.150 A 1.342 A 1.357 A 1.091 A	0.894 A 1.326 A 1.379 A 1.294 A 0.055 AE	NA	
	TIME 40 MINUTES	0.067 AE 0.072 AE 0.058 AE 0.035 AE 0.045 AE	0.074 AE 0.085 AE 0.166 AE 0.510 A 0.089 AE		
TEMP 90°F	TIME 25 MINUTES	NA		0.058 AE 0.087 AE 0.982 A 0.707 A 0.549 A	0.058 AE 0.038 AE 0.744 A 0.816 A 0.663 A
	TIME 40 MINUTES			0.072 AE 0.107 AE 0.078 AE 0.082 AE 0.056 AE	0.062 AE 0.042 AE 0.053 AE 0.062 AE 0.078 AE
TEMP 100°F	TIME 25 MINUTES	0.060 AE 0.101 AE 0.095 AE 0.038 AE 0.056 AE	0.074 AE 0.118 AE 0.155 AE 0.127 AE 0.069 AE	0 C 0.022 C 0.118 AE 0.004 C 0.065 C	0.070 C 0.040 C 0.029 C 0.090 C 0.077 C
	TIME 40 MINUTES	0.098 C 0.137 C 0.062 C 0.090 C 0.109 C	0.086 AE 0.090 AE 0.094 AE 0.073 AE 0.100 AE	0.027 C 0.017 C 0.025 C 0 C 0.067 C	0.030 C 0.007 C 0 C 0.031 C 0.006 C

THE NUMBERS INDICATE INCHES OF CRACK GROWTH WHEN EXPOSED TO 140°F AND 95-100% RELATIVE HUMIDITY FOR 1 HOUR

MATERIAL 2024-T3 BARE

WEDGE CRACK TEST

MODE OF FAILURE: A = ADHESIVE

C = COHESIVE 100 PERCENT

AE = SLIGHT ADHESIVE FAILURE AT EDGE OF SPECIMEN ONLY IN CRACK AREA

FIGURE 20. CHROMIC ACID ANODIZE MATRIX

2.6.3 (Cont'd)

The detailed racking, cleaning, deoxidizing procedures, plus the exact temperature, time, voltage and other data that were recorded while processing at each condition are reported in Appendix G.

The results of each condition tested in the anodizing matrix are shown in Figure 20.

The data shown in Figure 20 indicated that the best results, as far as adhesion was concerned, was obtained at high temperature (100°F), longer time (40 minutes), high concentration (8 oz/gal), and high voltage (23 volts). It was desired to determine if the range could be extended by increasing the temperature to 105°F at high voltage and high concentration. The results of those tests are shown in Figure 21. A review of the data, including tests from the sealing matrix, show that a workable range exists. The shaded blocks of Figure 21 show the proposed limits. The proposed range is narrow, but for use as a back up system, it is possible to use it both in the laboratory and in production. Further testing at a later date to expand the processing limits should be conducted.

2.6.3 (Cont'd)

TEMP	TIME	CONCENTRATION									
		6 OZ/GAL				7 OZ/GAL		8 OZ/GAL			
		17V		23V		20V		17V		23V	
85°	25	A	A	A	AE	-	-	-	-	-	-
	40	AE	AE	AE	AE	-	-	-	-	-	-
90°	25	-	-	-	-	-	-	A	AE	A	AE
	40	-	-	-	-	-	-	AE	AE	AE	AE
* 95°	35	-	-	-	-	C	C	-	-	-	-
100°	25	AE	AE	AE	AE	-	-	C	AE	C	C
	40	C	AE	AE	C	-	-	C	C	C	C
105°	25	-	-	-	-	-	-	AE	-	C	-
	40	-	-	-	-	-	-	AE	-	C	-
ALLOY		24	75	24	75	24	75	24	75	24	75

*FROM SEALING MATRIX

PROPOSED ANODIZE RANGE

95-100°F

7-8 OZ/GAL CONC.

20-23 VOLTS

35-40 MIN


A	ADHESIVE
AE	ADHESIVE EDGE
C	COHESIVE
-	NO TEST
	PROPOSED LIMITS

FIGURE 21. CHROMIC ACID ANODIZE MATRIX - WEDGE CRACK TESTS

2.7 Effects of Anodizing on Different Alloys and Tempers

Design of the final article had not progressed, at the beginning of the program, to the point of final selection of the alloys to be used. Several alloys and tempers had to be tested in the surface treatment investigation portion of the program to determine if there were any that would not perform satisfactorily in the processing procedure selected.

2.7 (Cont'd)

The following nonclad aluminum alloys were anodized using the median processing range (Table 1) of the phosphoric acid anodize process.

<u>Alloy</u>	<u>Temper</u>
7075	T73 and T76
7475	T6, T61, T731 and T761

One set (2 panels 6 x 6 x .125 inch) of each alloy and temper were bonded using the FM73/BR127 adhesive system. The bonded specimens were C-scan inspected and no voids were noted. The panels were then cut into wedge crack specimens and tested.

The results are shown in Figure 22. The results indicate that the choice of nonclad alloy and temper does not effect the bond durability produced by phosphoric acid anodize.

SPECIMEN NUMBER	CRACK EXTENSION (INCHES)	FAILURE MODE
7075-T76-1	0.065	COHESIVE
7075-T76-2	0.050	COHESIVE
7075-T76-3	0.070	COHESIVE
7075-T76-4	0.028	COHESIVE
7075-T76-5	0.075	COHESIVE
7075-T73-1	0.078	COHESIVE
7075-T73-2	0.022	COHESIVE
7075-T73-3	0.020	COHESIVE
7075-T73-4	0.058	COHESIVE
7075-T73-5	0.068	COHESIVE
7475-T731-1	0.084	COHESIVE
7475-T731-2	0.068	COHESIVE
7475-T731-3	0.060	COHESIVE
7475-T731-4	0.082	COHESIVE
7475-T731-5	0.045	COHESIVE
7475-T761-1	0.048	COHESIVE
7475-T761-2	0.033	COHESIVE
7475-T761-3	0.082	COHESIVE
7475-T761-4	0.102	COHESIVE
7475-T761-5	0.105	COHESIVE
7475-T61-1	0.058	COHESIVE
7475-T61-2	0.058	COHESIVE
7475-T61-3	0.055	COHESIVE
7475-T61-4	0.049	COHESIVE
7475-T61-5	0.087	COHESIVE

ENVIRONMENT EXPOSURE
(140°F 1 HOUR AT 95-100% RELATIVE HUMIDITY)

ADHESIVE SYSTEM BR 127 PRIMER

FM73 ADHESIVE FILM

FIGURE 22. BOND DURABILITY 7075/7475 ALUMINUM ALLOYS NONCLAD PHOSPHORIC
ACID ANODIZE WEDGE CRACK EXTENSION

SECTION III

CATHODIC PROTECTION OF LEAD IN PHOSPHORIC ACID

3. Introduction

During the early phosphoric acid anodizing runs a dark deposit was found on the sides and bottom of the anodizing tank. Sometimes portions of this loose spongy deposit could be seen suspended in the solution or floating on the surface. Analysis of the deposit showed it consisted primarily of lead with small amounts of copper and phosphorous.

A deposit containing heavy metals should not float in the phosphoric acid electrolyte but this deposit was spongy and probably contained appreciable quantities of trapped hydrogen.

After more use of the solution, white pearlescent needles were also found in the tank. Analysis of the needles showed them to be lead phosphates. Obviously the lead lining in the anodizing tank was being dissolved by the phosphoric acid electrolyte. Since the lead lining is the cathode during anodizing it was suspected that the attack was occurring when the tank was at rest. If this were true then cathodic protection should stop the attack of the tank lining and prevent the formation of both the dark, spongy deposit and the white needles.

3.1 Problem Analysis

A series of electrochemical measurements was made to determine critical potentials and to compare the electrochemical behavior of pure lead with lead antimony alloy (Pb 94%, Sb 6%).

Experiments were carried out in an 800 ml beaker containing 500 ml of dilute phosphoric acid solution at 73°F. The electrolyte contained 83.6 ml of 85% H_3PO_4 (Sp. Gr. 1.689) per liter. The electrodes used were:

- a. Chemically pure lead (Pb)
- b. Lead antimony alloy (Pb-Sb)
- c. Platinized titanium (Pt-Ti)
- d. Carbon (C)
- e. Saturated calomel electrode (SCE)

The Pb and Pb-Sb electrodes were cleaned, rinsed with distilled water, dried and weighed. At the conclusion of the study and several times during the experiments, the Pb and Pb-Sb electrodes were removed from the beaker, rinsed with distilled water, dried and reweighed. The electrodes did not lose weight. Only a minute weight gain was detected, indicating that either impurities in the electrolyte were being plated out or that a compound was being formed at the surface of the electrodes.

The following open circuit potentials were measured:

Pt-Ti vs. Pb	+0.73 V*
Pt-Ti vs. Pb-Sb	+0.79 V
Pb vs. Pb-Sb	+0.05 V
SCE vs. Pb	+0.15 V
SCE vs. Pb-Sb	+0.19 V
SCE vs. C	+0.49 V
C vs. Pb	+0.51 V
C vs. Pb-Sb	+0.55 V

Impressing a potential between the Pt-Ti or C electrodes and the Pb or Pb-Sb electrodes for a short time changed the open circuit potentials markedly.

* The (+) sign signifies that the first electrode is cathodic (more noble) than the second electrode.

Figures 23 and 24 illustrate the relationship between impressed voltage and current density for Pt-Ti electrodes vs. Pb and Pb-Sb respectively. While the zero-current potential is about the same for both alloys (+1.8 volts), the behavior in the anodic region is substantially different for the two alloys.

At a potential of 2.1 to 2.2 volts, slight gassing was observed on the Pb and Pb-Sb cathodes. Thus a potential of about 1.8 to 1.9 volts should be maintained between the Pt-Ti anode and the Pb-Sb cathode.

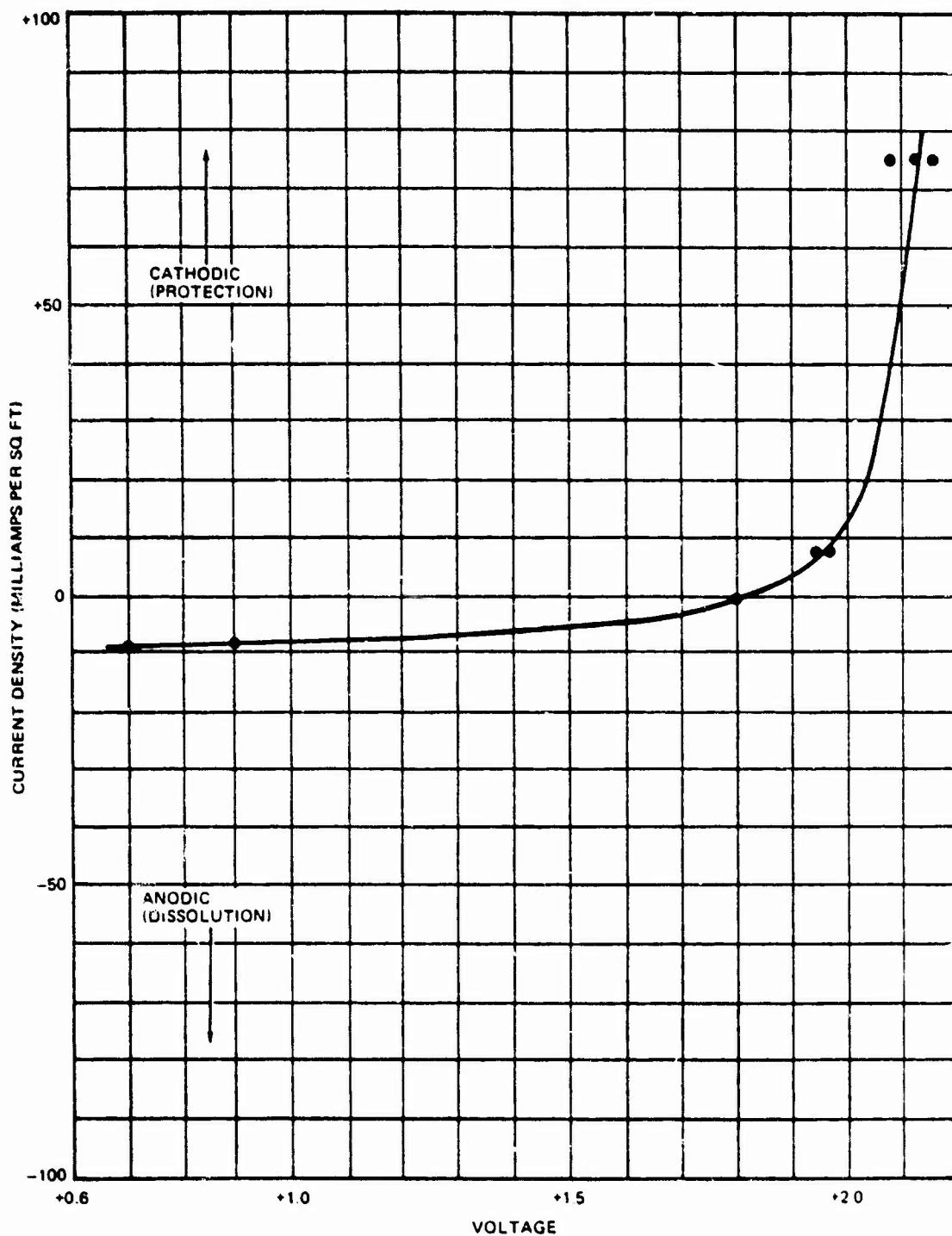


FIGURE 23. CATHODIC PROTECTION OF PURE LEAD USING PLATINIZED TITANIUM

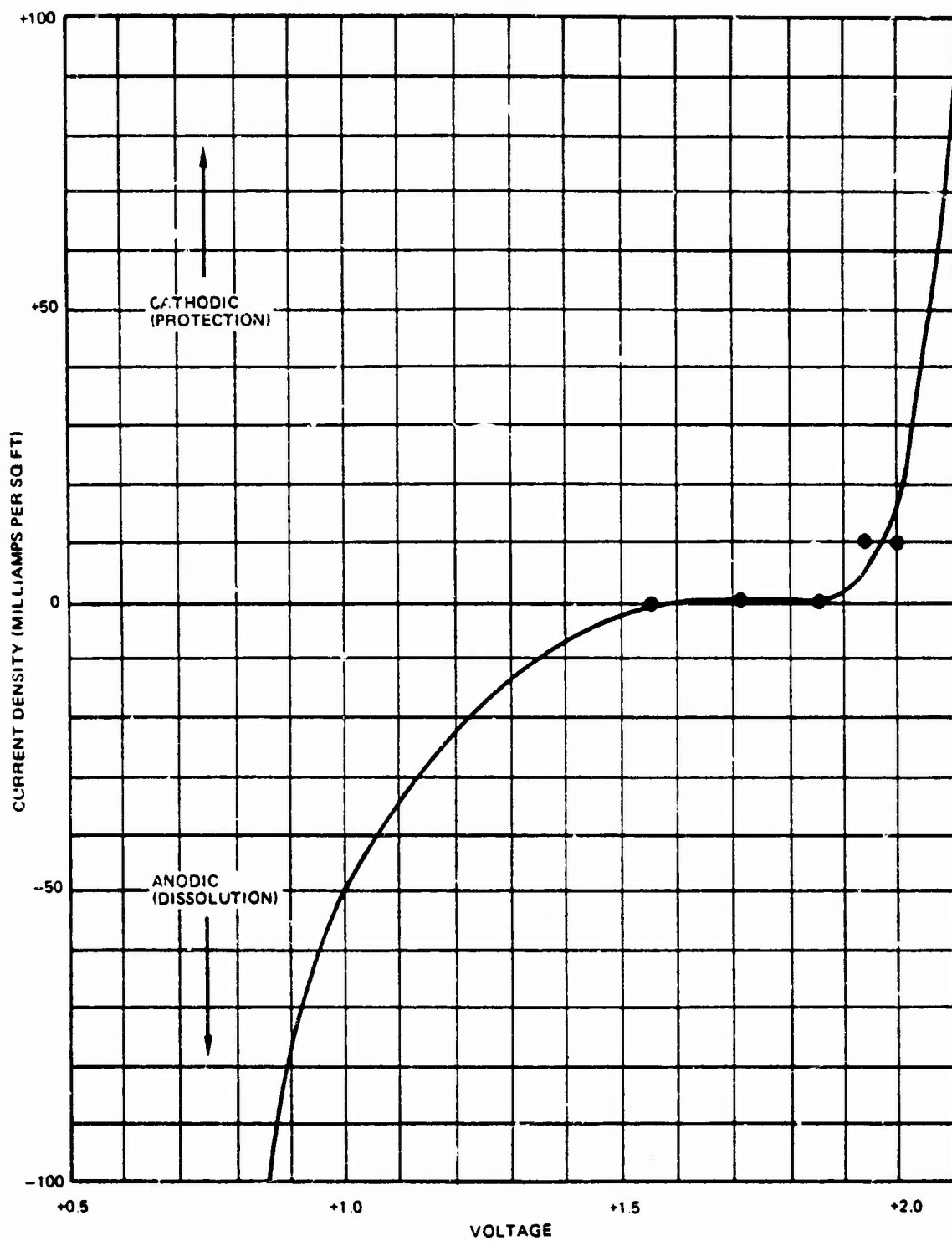


FIGURE 24. CATHODIC PROTECTION OF LEAD-ANTIMONY USING PLATINIZED TITANIUM

Measurements were also made using a carbon anode in the hope that the cost of a platinized electrode in a large anodizing tank could be avoided. The zero-current potential for the C, Pb-Sb couple varied between 1.3 and 1.5 volts. A 7 liter pilot tank was set up using a carbon anode and a Pb-Sb cathode in a 13.5 oz/gal H_3PO_4 electrolyte. A potential of 1.75 to 1.80 volts was applied for one week and at the end of this time it was noticed that the solution had darkened. Other types of carbon anodes were subsequently tried with fresh H_3PO_4 electrolytes but the result was always the same: darkening of the solution until it eventually became coffee-colored.

A fresh H_3PO_4 electrolyte was prepared and a large Pt-Ti anode was used with a Pb-Sb cathode. A potential between 1.8 and 2.1 volts was applied. At the end of one week it was noticed that a gelatinous substance was present on the anode.

X-ray fluorescence analysis of the substance revealed platinum with no other elements detected.

The platinized titanium anode also changed color. This was due to the dissolution of platinum in the phosphoric acid electrolyte and the subsequent anodizing of the exposed titanium. Even though a satisfactory anode material has not been found, the use of cathodic protection for the lead alloy tank lining has been effective to prevent attack and formation of deposits.

3.2 Dissolution Rate of Lead and Lead-Antimony Alloy in Phosphoric Acid

3.2.1 Introduction

The tank lining material for the principal laboratory phosphoric acid anodizing tank is pure lead and a lead 6% antimony alloy for the secondary tank. The production tank lining is constructed with this same lead (Pb-Sb) alloy.

The following test was initiated to gauge the rate of this dissolution in phosphoric acid solution.

3.2.2 Procedure

3.2.2.1 Pure Lead Material in Phosphoric Acid Solution

- a. Three pieces, one inch square, were cut from a pure lead sheet.
- b. After cleaning and oven drying, the specimens when cooled were weighed and measured.
- c. The specimens were suspended by a No. 20 four cord cotton thread in a glass beaker containing 2000 ml of the anodizing solution prepared at a concentration of 16 oz/gal of 85% phosphoric acid.
- d. The beaker was covered with a plastic sheeting to minimize evaporation and was set aside in the laboratory.
- e. Following an exposure period, the specimens were removed, rinsed, scrubbed with a soft bristle brush to remove any loose reaction products and oven dried.
- f. The specimens were reweighed and remeasured.
- g. After recording the changes, the specimens were re-immersed in acid solution for the next exposure period.
- h. From the repetition of this re-immersing procedure, the findings were translated cumulatively in the graph of Figure 25 with the dissolution rate computed as inches of material lost projected for a one year span.

3.2.2.2 Lead 5% Antimony Alloy Material in Phosphoric Acid Solution

- a. Three pieces, one inch square, were cut from an excess portion of the lead-antimony alloy plate material being used in the construction of the lining for the production anodizing tank.
- b. The specimens were treated and tested in the identical procedures as with the pure lead specimens.

3.2.2.3 Results

The material loss in inches of the respective test specimens are presented in Figure 25, together with projections of such losses for a span of one year. It is apparent that the pure lead liner has the lowest dissolution rate in the tests performed. Other liners have been used in industry, including stainless steel and polymer types. These systems were not tested in this program. Prior to the installation of a tank system for production, a thorough analysis should be made to determine which type of liner is most suitable for the system being installed.

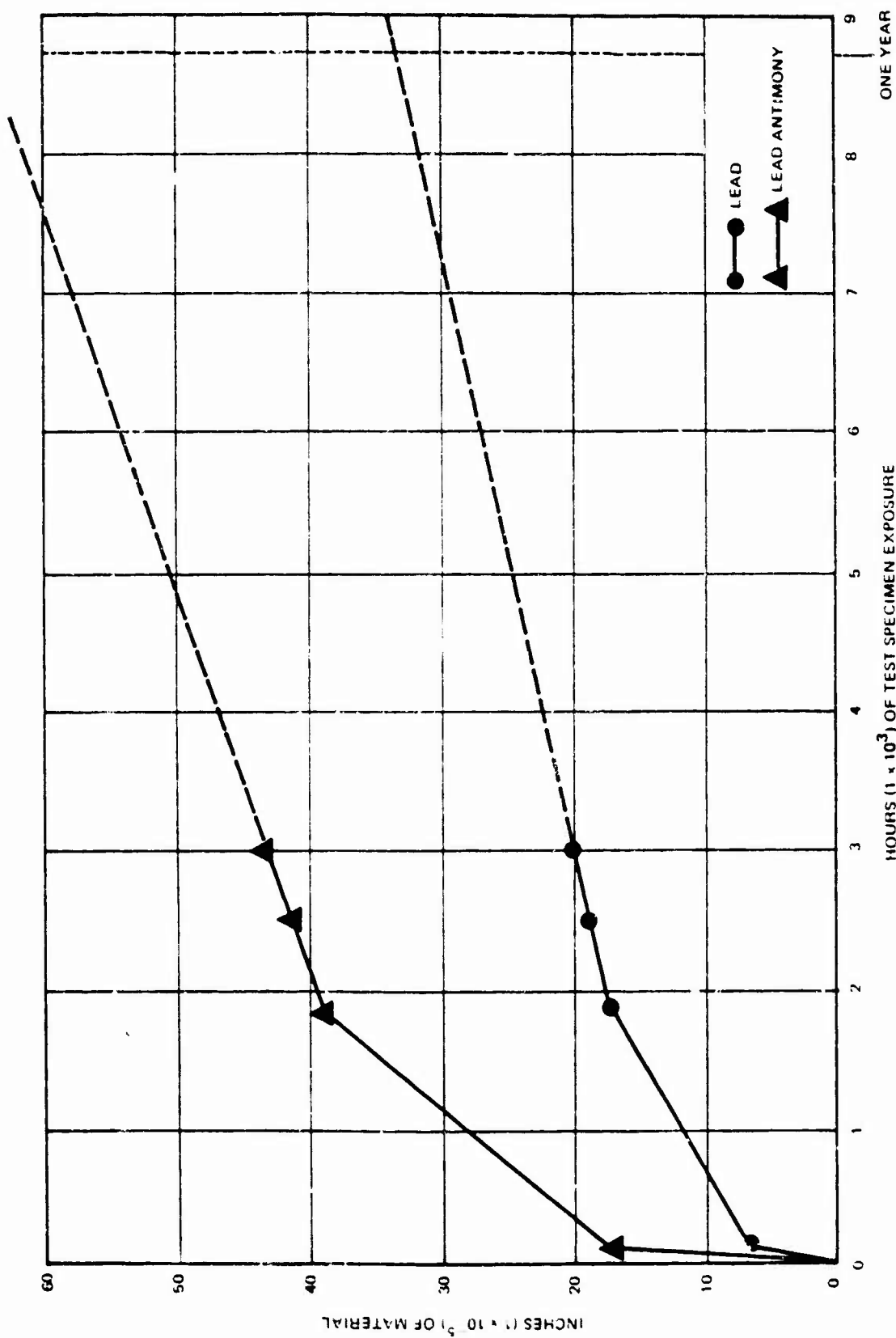


FIGURE 25. LEAD EXPOSURE TO PHOSPHORIC ACID

SECTION IV

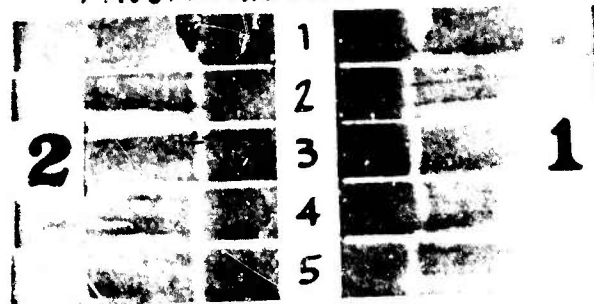
SURFACE TREATMENT CONTAMINATION

4. Introduction

Two types of contamination have been experienced in the production anodize system that are considered significant. One is the growth, in the phosphoric acid anodize solution, of black mold which has not caused problems with the efficiency of the anodizing process and can be effectively filtered from the solution. The second type of contamination is associated with a mechanical destruction of the anodize surface by handling. This type of contamination can be experienced on other types of anodize and surface treatments. This section of this report deals with the investigation of this second type of contamination.

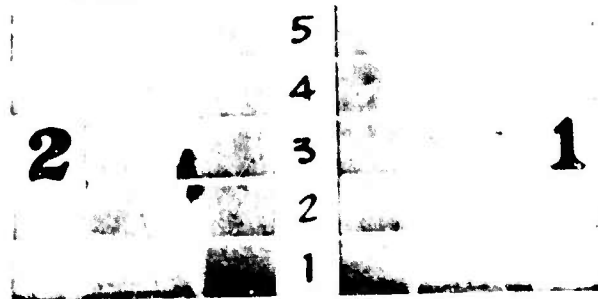
Production load test failures of two sets of anodized wedge crack test specimens (Figure 26) initiated an investigation of materials commonly used in handling metal bond details. These failed wedge crack test specimens were made from two 0.125 x 6 x 6 inch plates of 7075-T6 aluminum. These specimens were alkaline cleaned, acid etched, and phosphoric acid anodized for 22 minutes at 10 volts in 8% by weight H_3PO_4 . After oven drying, following rinsing, in the production facility, a QA check for anodize was made and parts accepted. The panels were then removed from the anodizing rack using white gloves, wrapped in kraft paper, and transported (hand-carried 300 yards) to the laboratory. Within 4 hours the panels were primed with a 0.0001 to 0.0002 inch coat of BR127 adhesive primer which was cured for 30 minutes at room temperature plus 60 minutes at 250°F. The panels were bonded together using Narmco M1133 adhesive and cured for 90 minutes at 245°F. Five wedge crack specimens were cut from this assembly. The wedge crack test specimens were exposed for 3 hours at 140°F, 100% RH using a 0.125 inch wedge. Adhesive failure was observed for the first time on phosphoric acid anodized parts. Since these parts were removed from the racks before priming, contamination was suspected.

PHOSPHORIC ACID ANODIZE



7612 PRODUCTION ANODIZE - LAB PRIME

PHOSPHORIC ACID ANODIZE



7612 PRODUCTION ANODIZE - LAB PRIME

FIGURE 26. PRODUCTION ANODIZE WEDGE CRACK FAILURE

4.1 Abrasion - Compression

The effect of abrasion and compression was investigated by scanning electron microscope (SEM) as shown in Figure 27. A glass stirring rod, drawn across the surface of a phosphoric acid anodized panel, caused a furrow down to bare metal with residue being thrown out on the compressed surface adjacent to it. The compression area is readily seen when clean etched aluminum foil is placed over the phosphoric acid anodized surface and a glass stirring rod or a hard plastic roller is used to cause the compression.

The test was devised to cause compression damage of phosphoric acid anodize to segregate physical damage from the effects of contamination which may incur some physical damage.



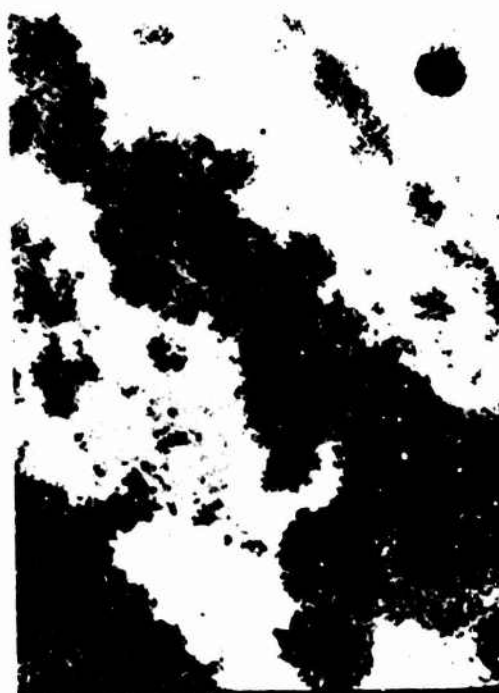
GLASS ROD

6000X



FOIL-GLASS

15,000X



ALUMINUM FOIL - ROLLER

5000X



ALUMINUM FOIL - ROLLER

25,000X

FIGURE 27. PHOSPHORIC ACID ANODIZE ABRASION-COMPRESSION

4.2 Contamination Test Procedure

The program was further expanded to determine effects of the same types of contamination on panels treated with (1) FPL etch and (2) potassium dichromate sealed chromic acid anodize and (3) phosphoric acid anodize. Uncontaminated panels and those panels damaged by a hard plastic roller over aluminum foil were made into control specimens. Others were contaminated by white cotton gloves, clean kraft paper, or bare hands. The wedge crack test specimens used were 0.125- x 6- x 6-inch plates of 7075-T6 nonclad aluminum. The panels were surface treated, contaminated by lightly rubbing the bond surface of the No. 1 panel with the specified contaminants (except for the uncontaminated and the aluminum foil compression specimens). These panels were primed within 2 hours with American Cyanamid BR127, air dried for 30 minutes, and then baked for 1 hour at 250°F. All the panels were bonded using Namco M1133 adhesive and cured for 90 minutes at 245°F and 40 psi.

Phosphoric acid anodized panels were processed as follows:

- a. Solvent cleaned
- b. Alkaline cleaned
- c. Deoxidized
- d. Phosphoric acid anodized for 22 minutes at 10 volts in 8% by weight solution of H_3PO_4 .

FPL etched panels were processed as follows:

- a. Solvent cleaned
- b. Alkaline cleaned
- c. FPL etched for 15 minutes at 160°F in an FPL etch bath modified by addition of 2024 aluminum alloy.

Chromic acid anodized panels were processed as follows:

- a. Solvent cleaned
- b. Alkaline cleaned
- c. Deoxidized
- d. Chromic acid anodized for 35 minutes at 21.5 volts in a 7.5 oz/gal chromic acid solution.
- e. Sealed 10 minutes at 190°F in a potassium dichromate solution (5% by weight) using deionized water with a pH of 4.0.

4.3 Phosphoric Acid Anodize

There were no wedge crack failures in the phosphoric acid anodized surface control specimens or the aluminum foil compressed surface specimens. The white cotton glove, kraft paper, and bare hand contaminated specimens showed gross adhesive failure (See Figure 28).

Figures 29a to 29d SEM micrographs show the phosphoric acid anodize surfaces before priming.

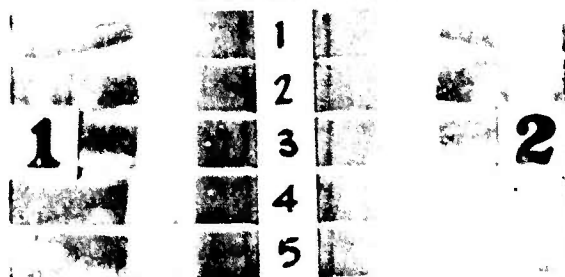
Figure 30 shows the SEM micrograph of the bare metal side of the cotton glove contaminated wedge crack failure. Note adhesive in some of the pits and grain boundaries where there was no contamination.

Figure 31 shows the SEM micrograph of the adhesive side of the cotton glove contaminated wedge crack failure. Note the replication of grain boundaries, pits, and the phosphoric acid anodize itself.

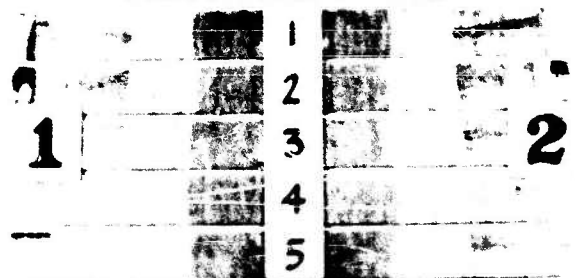
The Auger spectra of the surface before priming (Figure 32) and the carbon depth profile (Figure 33) show the great increase in carbon from any of the contamination methods.

Compression of phosphoric acid anodize in a hydraulic press using aluminum foil and kraft paper even at 100,000 psi, which caused great deformation of the anodize film, did not result in any wedge crack failure (Figure 34). The SEM micrograph of the aluminum foil compression is shown in Figure 35.

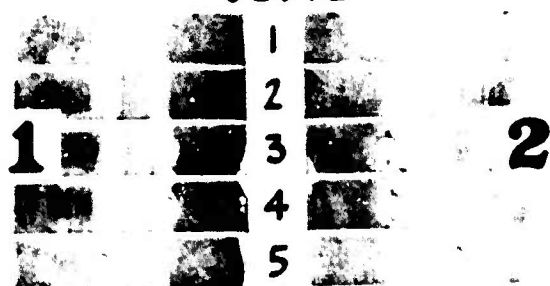
PHOSPHORIC ACID ANODIZE CONTROL



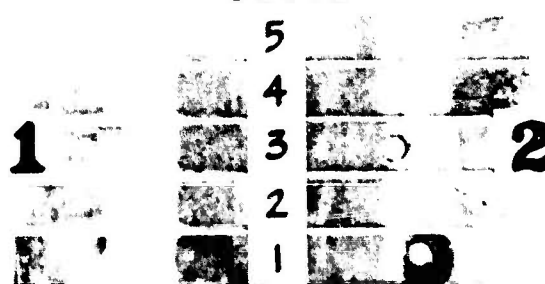
PHOSPHORIC ACID ANODIZE ALUMINUM FOIL



PHOSPHORIC ACID ANODIZE GLOVE

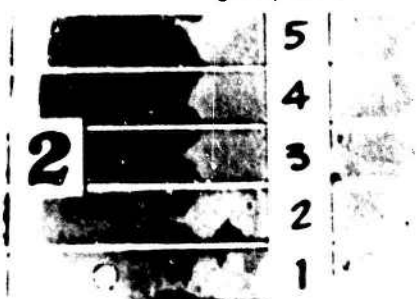


PHOSPHORIC ACID ANODIZE GLOVE



NO PRIMER

PHOSPHORIC ACID ANODIZE GLOVE



PHOSPHORIC ACID ANODIZE KRAFT PAPER

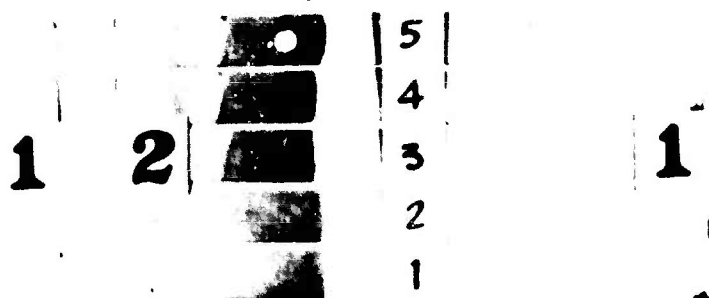


FIGURE 28. PHOSPHORIC ACID ANODIZE WEDGE CRACK

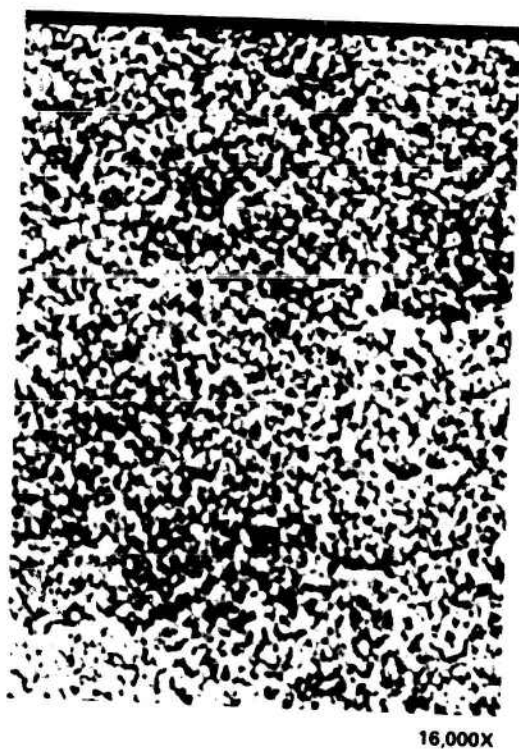


FIGURE 29a. UNCONTAMINATED CONTROL PHOSPHORIC ACID ANODIZE – SEM



FIGURE 29b. ALUMINUM FOIL COMPRESSED PHOSPHORIC ACID ANODIZE - SEM



800X



4000X



16,000X



27,000X

FIGURE 29c. COTTON GLOVE CONTAMINATED PHOSPHORIC ACID ANODIZE – SEM



800X



4000X

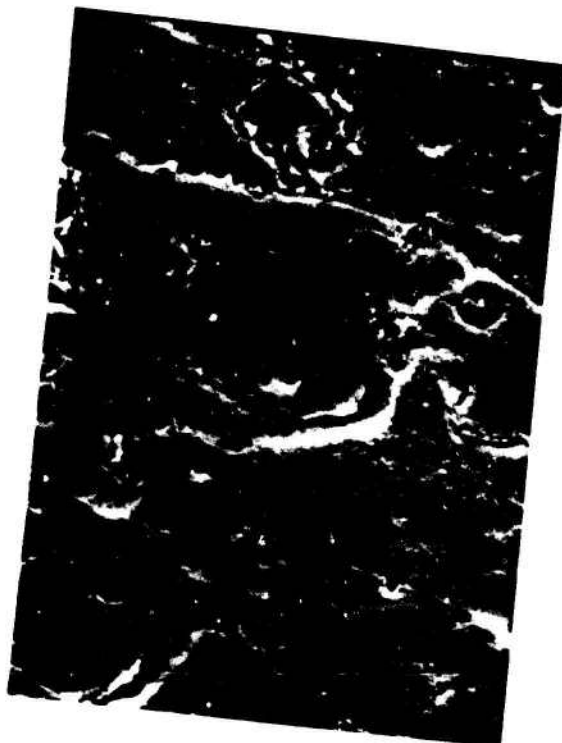


16,000X

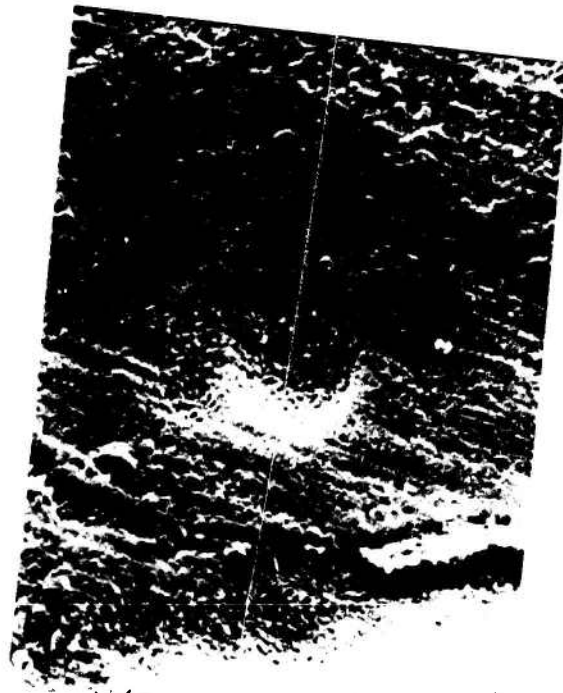


25,000X

FIGURE 29d. KRAFT PAPER CONTAMINATED PHOSPHORIC ACID ANODIZE - SEM



800X

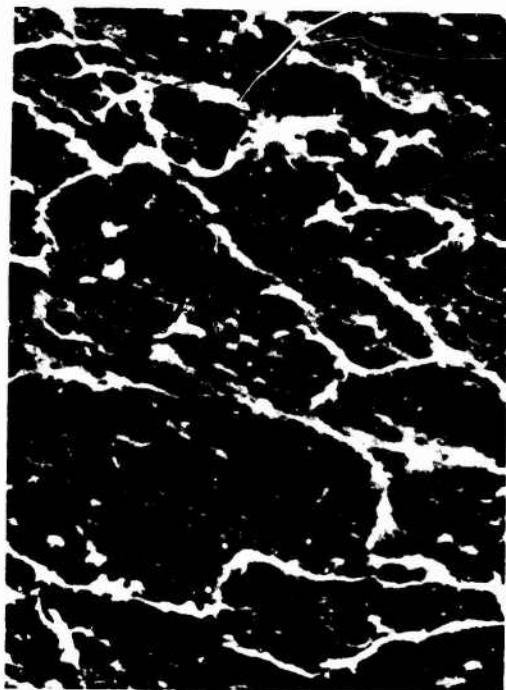


4000X



16,000X

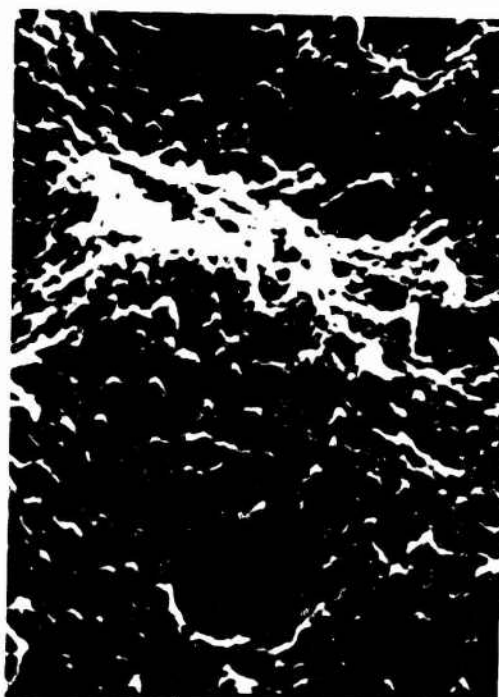
FIGURE 30. COTTON GLOVE CONTAMINATED PHOSPHORIC ACID ANODIZE BARE METAL.
WEDGE CRACK FAILURE



800X



4000X



16,000X

FIGURE 31. COTTON GLOVE CONTAMINATED PHOSPHORIC ACID ANODIZE ADHESIVE
WEDGE CRACK FAILURE

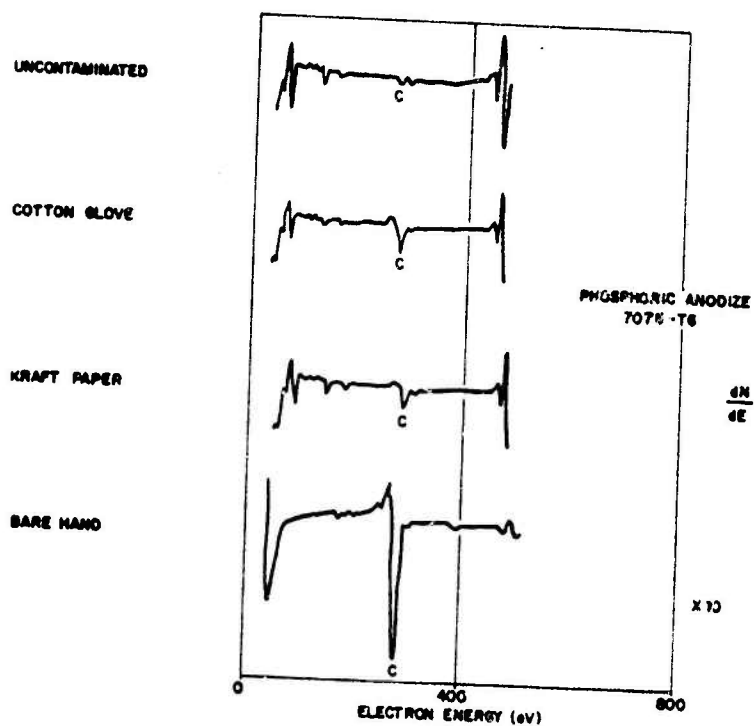


FIGURE 32. AUGER SPECTRA

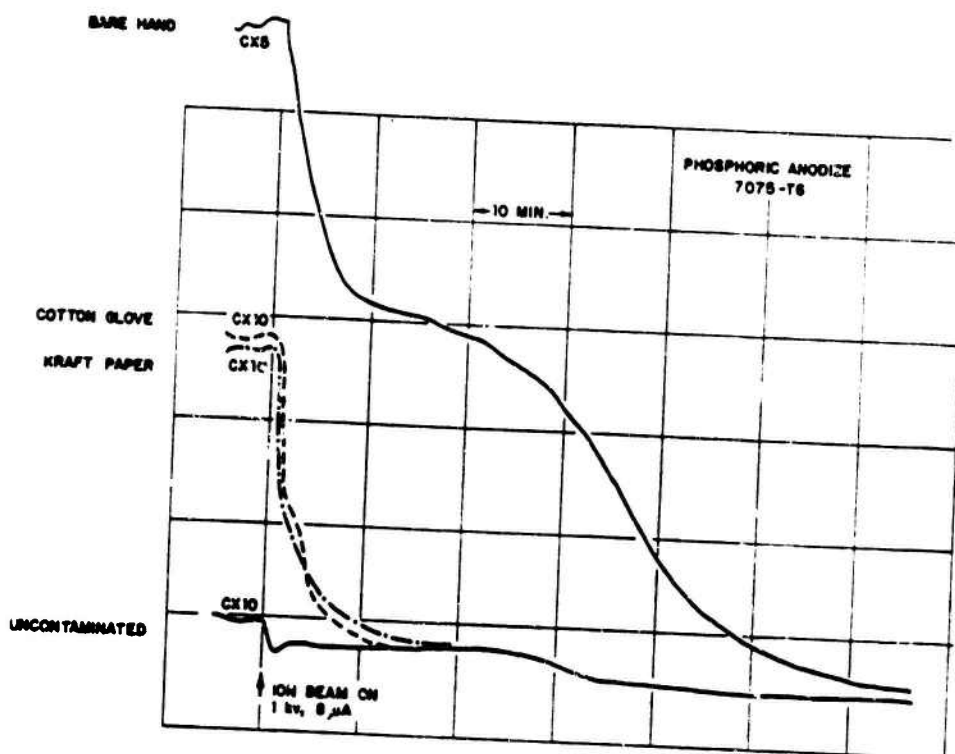
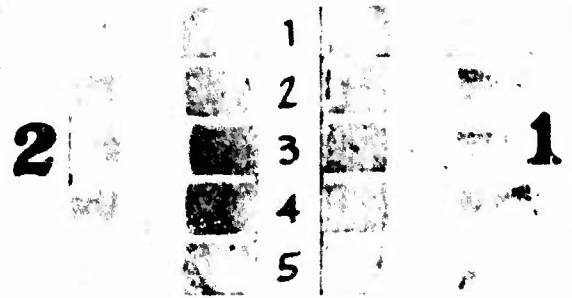


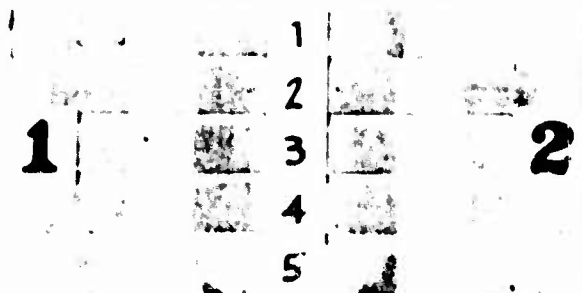
FIGURE 33. CARBON DEPTH PROFILE

PHOSPHORIC ACID ANODIZE
ALUMINUM FOIL



HYDRAULIC PRESS
100,000 PSI

PHOSPHORIC ACID ANODIZE
KRAFT PAPER



HYDRAULIC PRESS
100,000 PSI

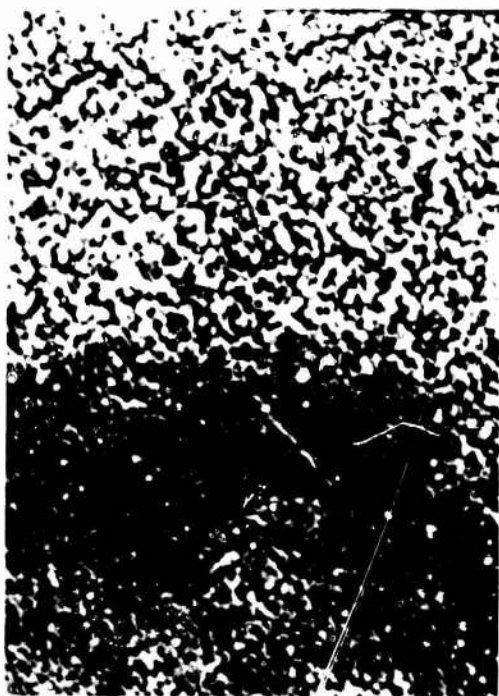
FIGURE 34 HYDRAULIC PRESS CONTAMINATION



800X



5000X



16,000X



26,000X

FIGURE 35. ALUMINUM FOIL HYDRAULIC PRESS - SEM

4.4 FPL Etch

Figure 36 shows very slight edge failure in the uncontaminated control and the aluminum foil compressed specimens. This type of failure is typical of FPL etched wedge crack specimens. Gross failure is shown with FPL etched specimens contaminated with white cotton gloves, kraft paper, or bare hands.

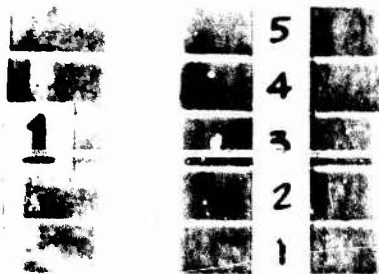
SEM micrographs, Figures 37a through 37e, show the FPL etched specimens before priming.

Figure 38 shows the metal side of the wedge crack failure specimen contaminated with cotton glove and Figure 39 with kraft paper. Note the undisturbed normal micropitting of FPL etch.

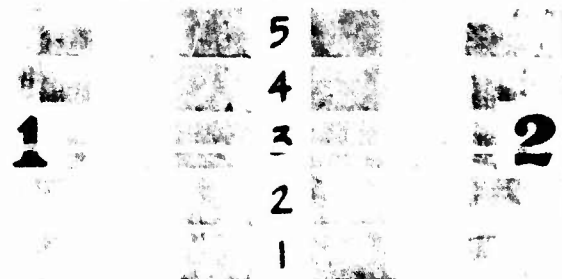
Figure 40 shows the adhesive side of the wedge crack failure specimen contaminated with cotton glove and Figure 41 with kraft paper. Note the replication of grain boundaries, pits, and micropitting.

Auger spectra of the surface before priming (Figure 42) and the carbon depth profile (Figure 43) show the increased carbon present.

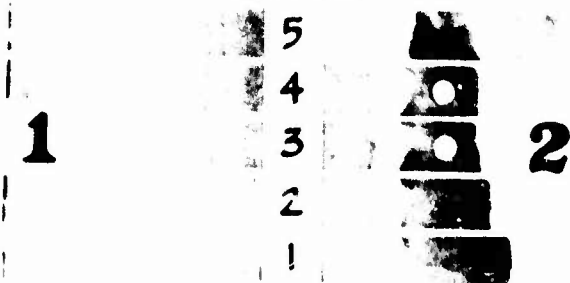
FPL ETCH CONTROL



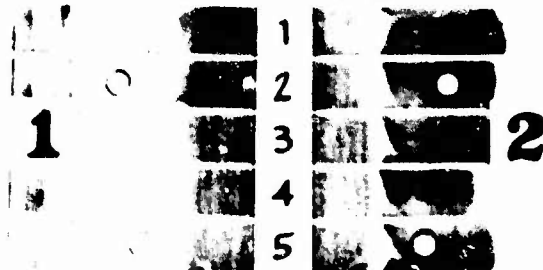
FPL ETCH ALUMINUM FOIL



FPL ETCH GLOVE



FPL ETCH KRAFT PAPER



FPL ETCH HAND

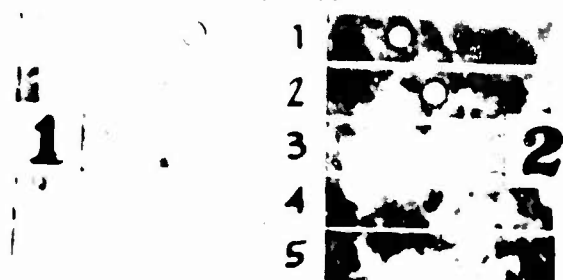
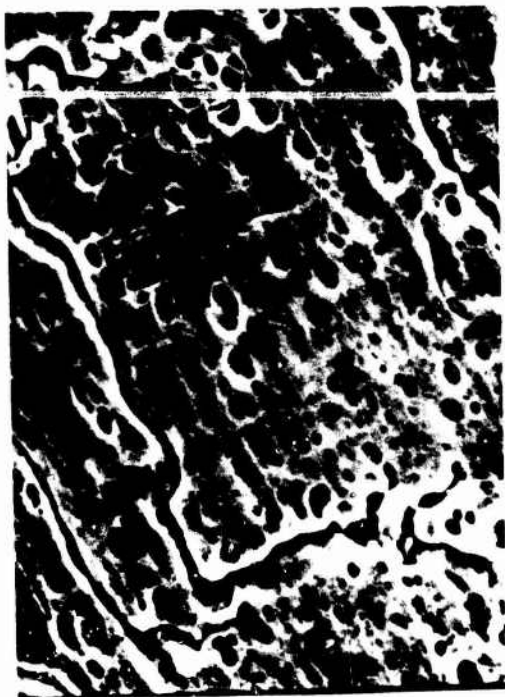


FIGURE 36. FPL WEDGE CRACK TEST



800X



4000X



16,000X



27,000X

FIGURE 37a. UNCONTAMINATED CONTROL FPL ETCH - SEM



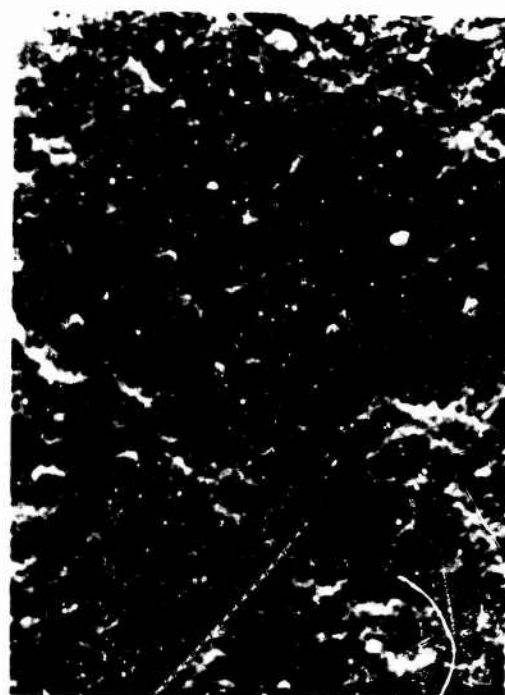
800X



4000X



16,000X

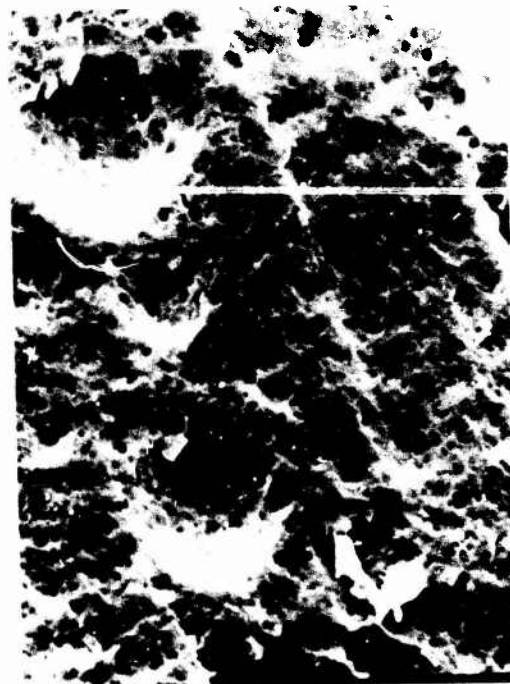


27,000X

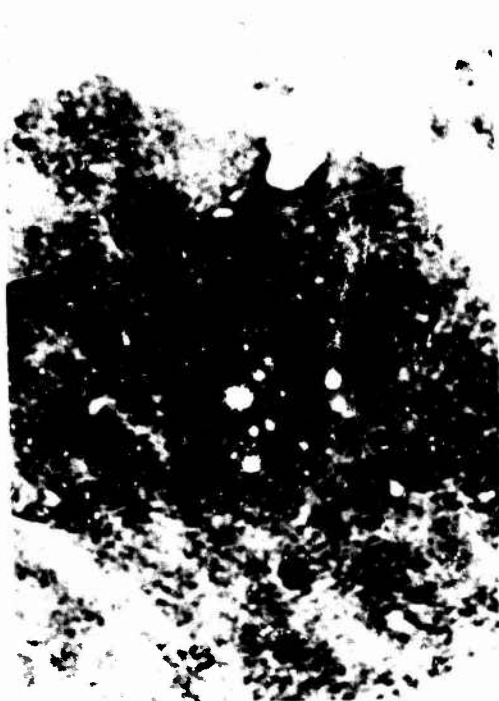
FIGURE 37b. ALUMINUM FOIL COMPRESSED FPI ETCH - SEM



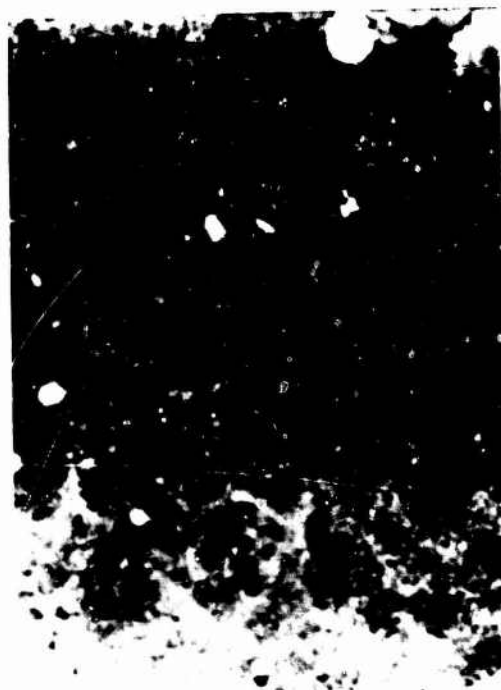
800X



4000X



18,000X



25,000X

FIGURE 37c. COTTON GLOVE CONTAMINATED FPL ETCH - SEM



800X



4000X



16,000X



27,000X

FIGURE 37d. KRAFT PAPER CONTAMINATED FPL ETCH - SEM



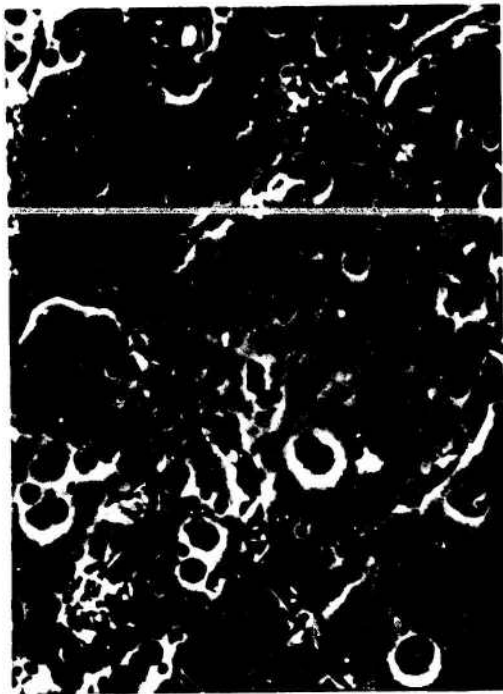
X000A



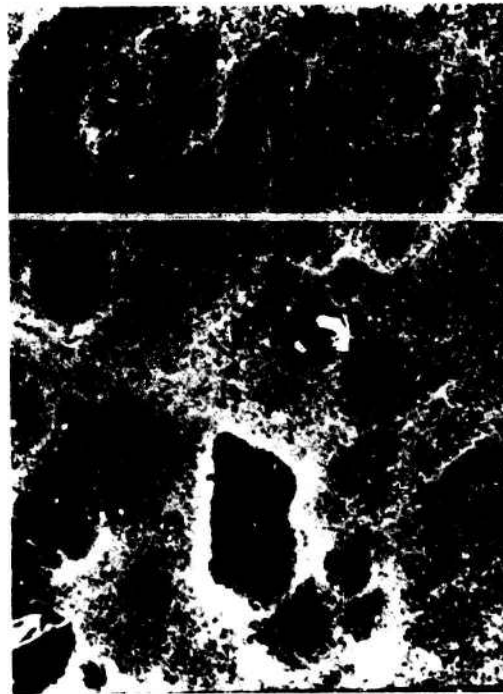
X008



37 BARE HAND CONTAMINATED FPL E



800X



4000X



16,000X

**FIGURE 38. COTTON GLOVE CONTAMINATED FPL ETCHED NONCLAD METAL WEDGE
CRACK FAILURE**

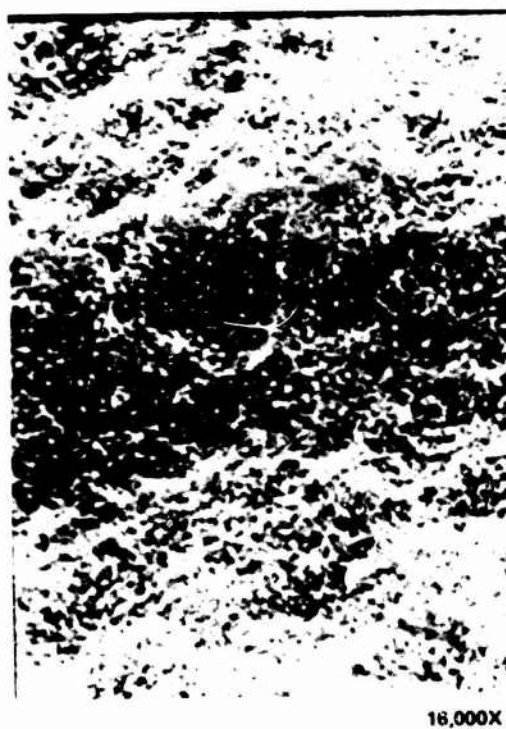
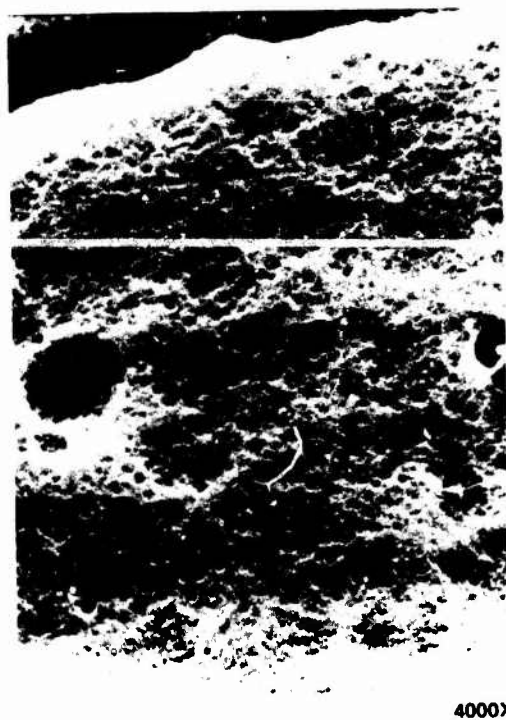
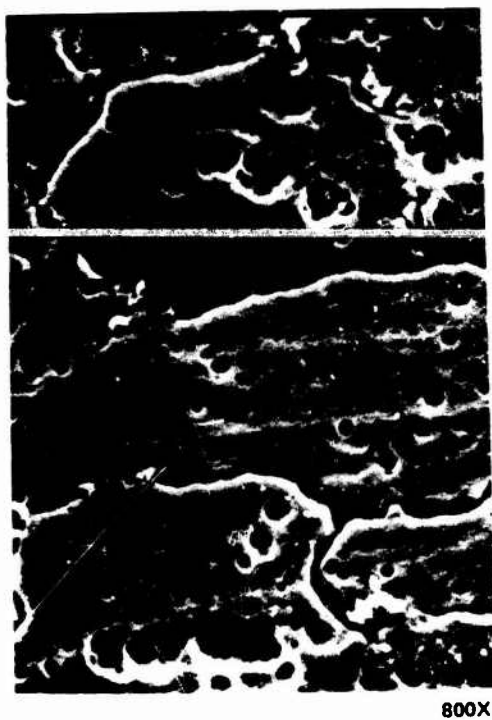
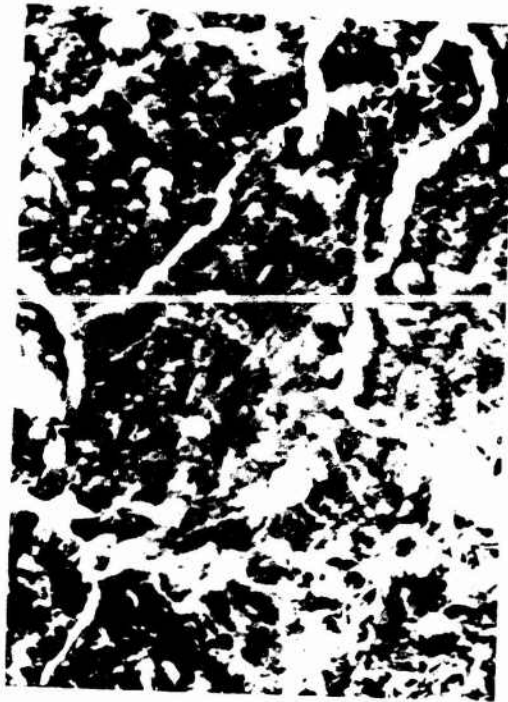


FIGURE 39. KRAFT PAPER CONTAMINATED FPL ETCHED ADHESIVE WEDGE CRACK FAILURE



800X

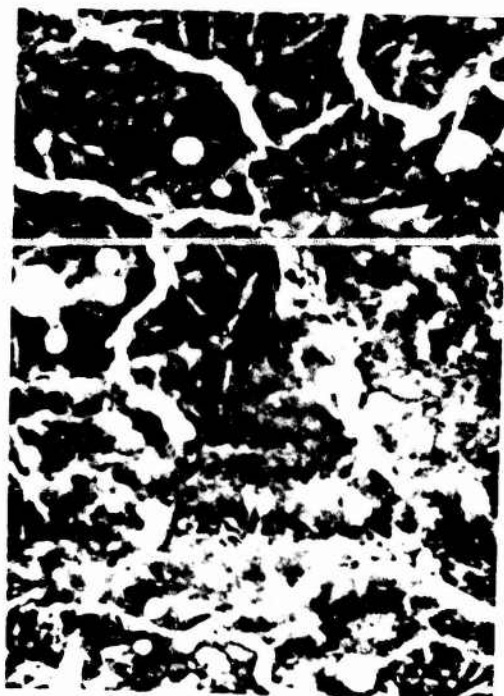


4000X



16,000X

FIGURE 40. COTTON GLOVE CONTAMINATED FPL ETCHED ADHESIVE WEDGE CRACK FAILURE



800X



4000X



16,000X

FIGURE 41. KRAFT PAPER CONTAMINATED FPL ETCHED ADHESIVE WEDGE CRACK FAILURE

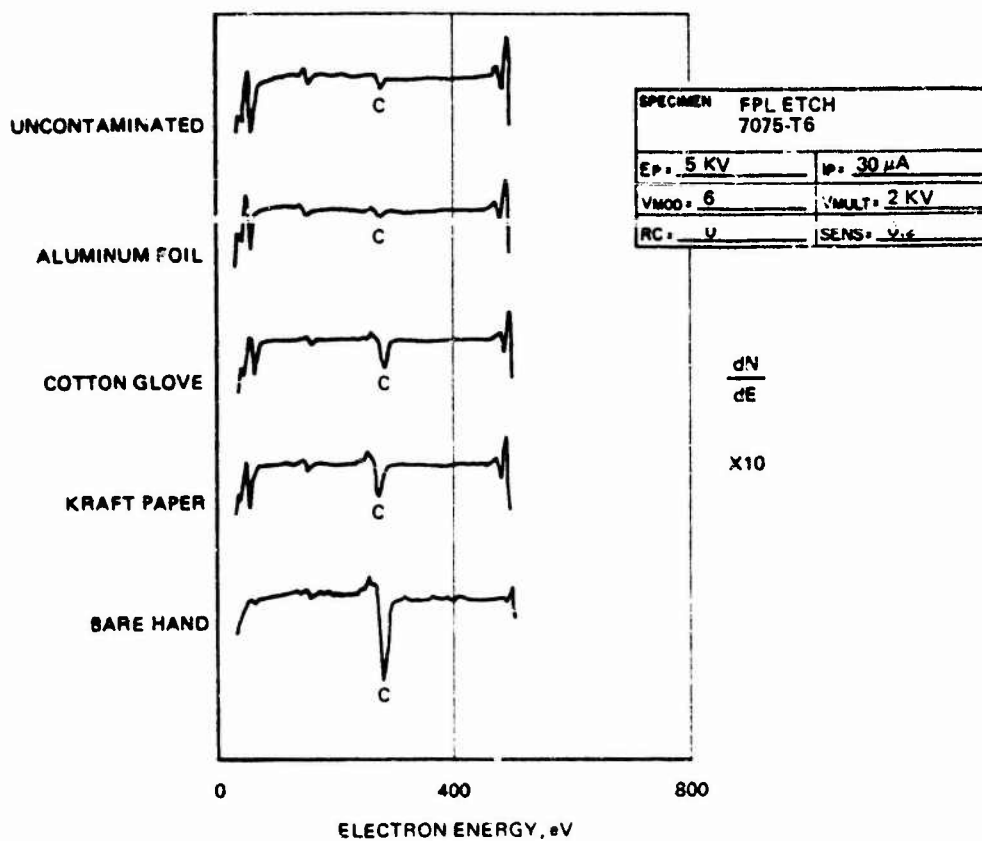


FIGURE 42. FPL - AUGER SPECTRA

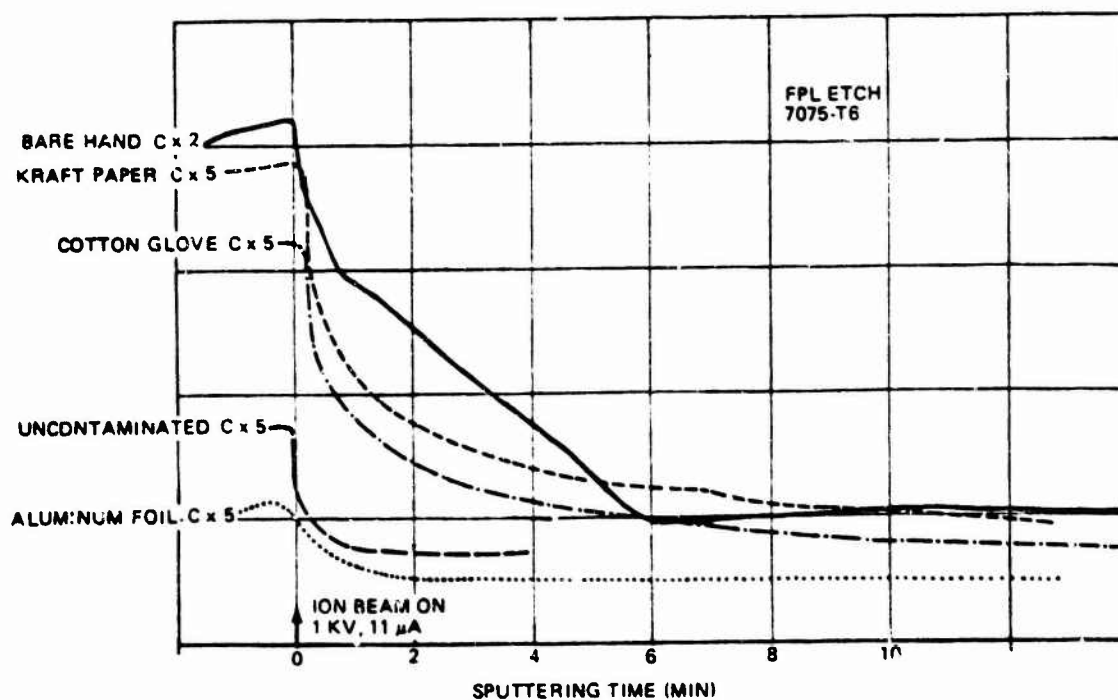


FIGURE 43. FPL - CARBON DEPTH PROFILE

4.5 Chromic Acid Anodize

Figure 44 shows the wedge crack tests of the sealed chromic acid anodized surfaces. There were no adhesive failures of the control or aluminum foil compressed surface specimens. Adhesive failure of the wedge-crack test specimen was evident only in the area where they were contaminated with cotton gloves, kraft paper, and bare hands.

NOTE: As the anodized surface is gray-green in color, the failure area appears as a gray streak within the white stressed adhesive area exposed in the wedge crack humidity test.

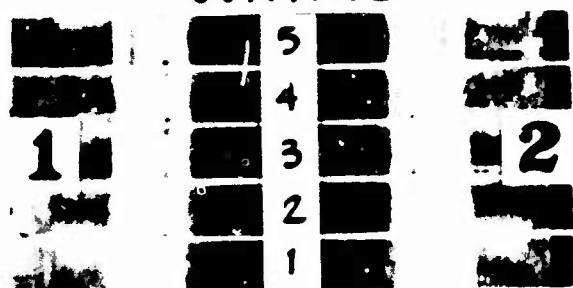
Figures 45a through 45e show the SEM micrographs of the chromic acid anodized surfaces before priming.

Figure 46 shows the bare metal side of the bare hand contaminated wedge crack failure. The lower half of the low-magnification SEM micrograph shows adhesive failure. The upper half of the low-magnification micrograph and the other two micrographs show fracture within the anodic film.

Auger spectra of the surfaces before priming (Figure 48) and the carbon depth profile (Figure 49) show only a small increase in carbon from contamination. There was a large increase in carbon for bare hand contamination of the chromic acid anodized surface.

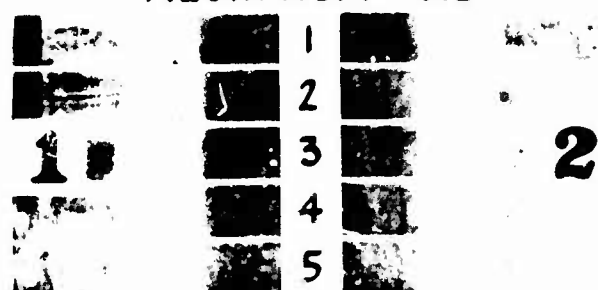
CHROMIC ACID ANODIZE

CONTROL



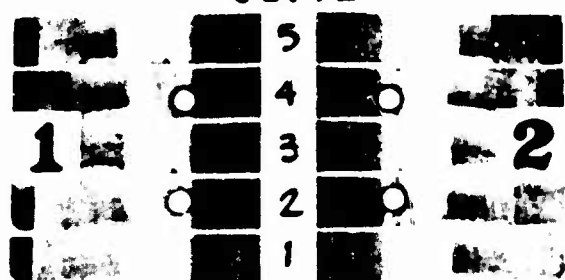
CHROMIC ACID ANODIZE

ALUMINUM FOIL



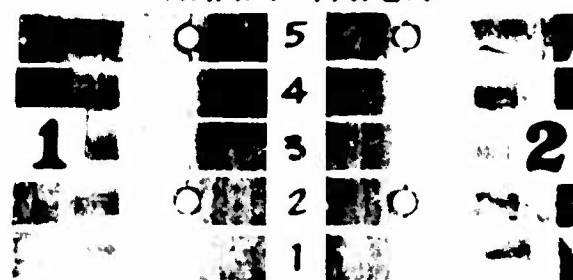
CHROMIC ACID ANODIZE

GLOVE



CHROMIC ACID ANODIZE

KRAFT PAPER



CHROMIC ACID ANODIZE

HAND

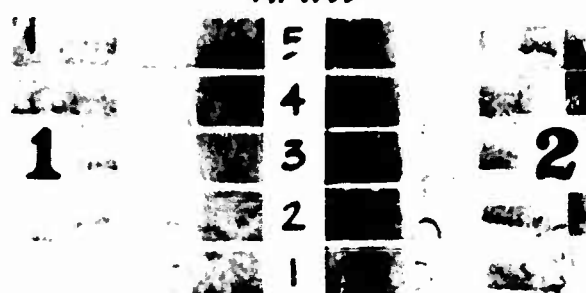


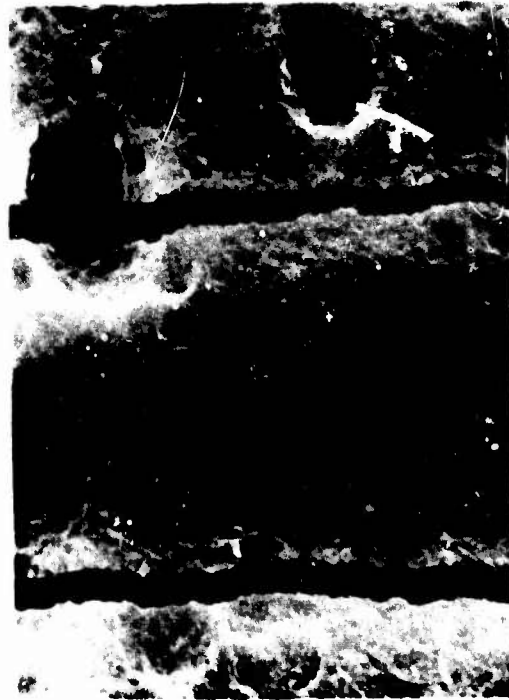
FIGURE 44. CHROMIC ACID ANODIZE - WEDGE CRACK TEST



FIGURE 45a. CONTROL UNCONTAMINATED CHROMIC ACID ANODIZE - SEM



800X



4000X



15,000X



10,400X

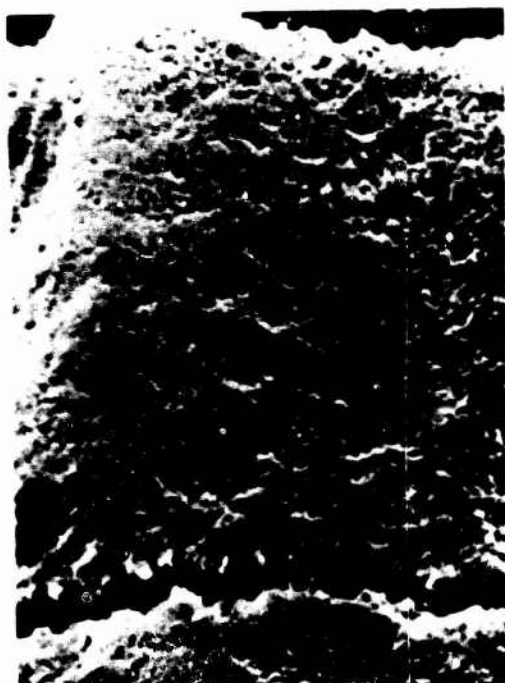
FIGURE 45b. ALUMINUM FOIL COMPRESSED CHROMIC ACID ANODIZE - SEM



800X



4000X



16,000X

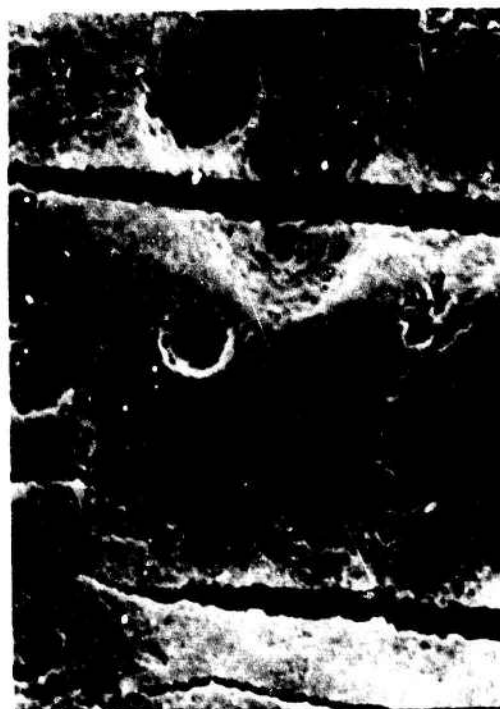


70,000X

FIGURE 45c. COTTON GLOVE CONTAMINATED CHROMIC ACID ANODIZE — SEM



800X



4000X



16,000X



10,800X

FIGURE 45d. KRAFT PAPER CONTAMINATED CHROMIC ACID ANODIZE - SEM

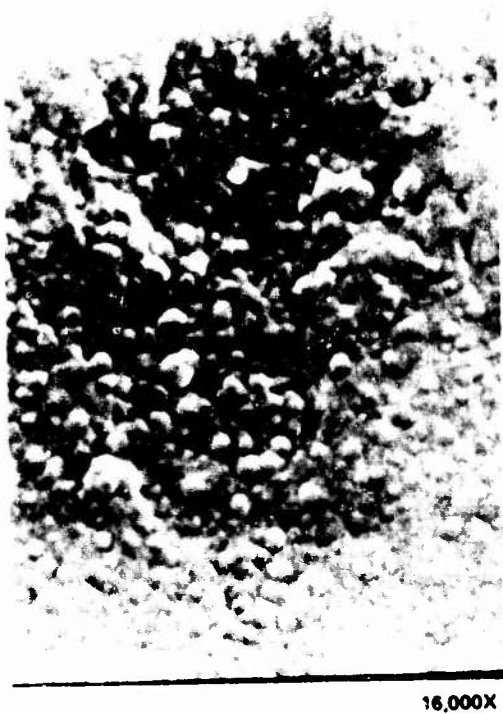
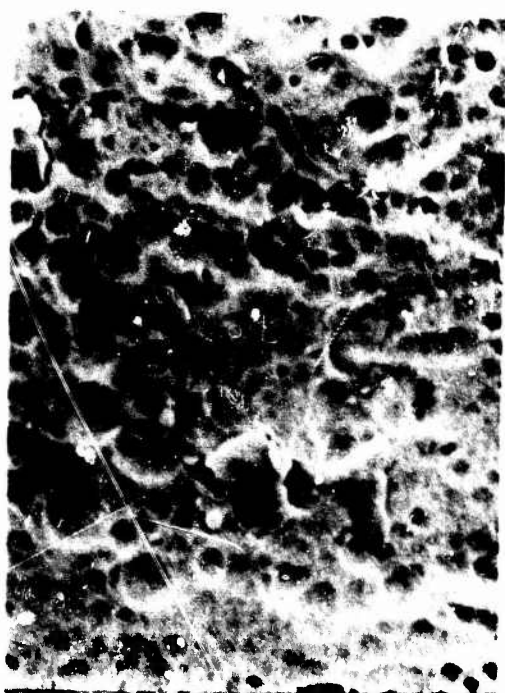
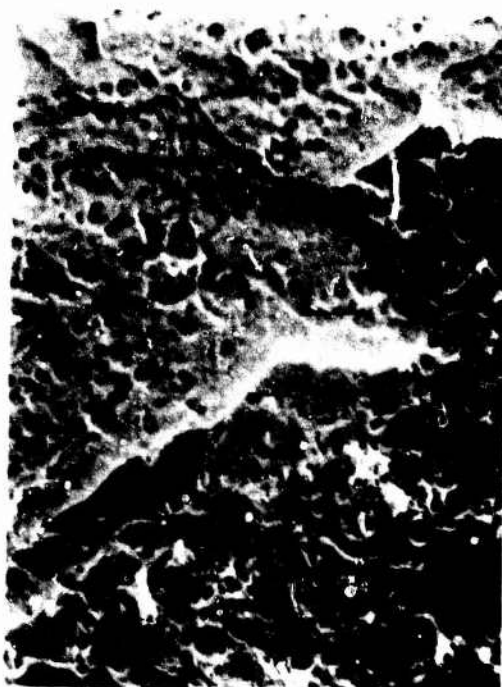


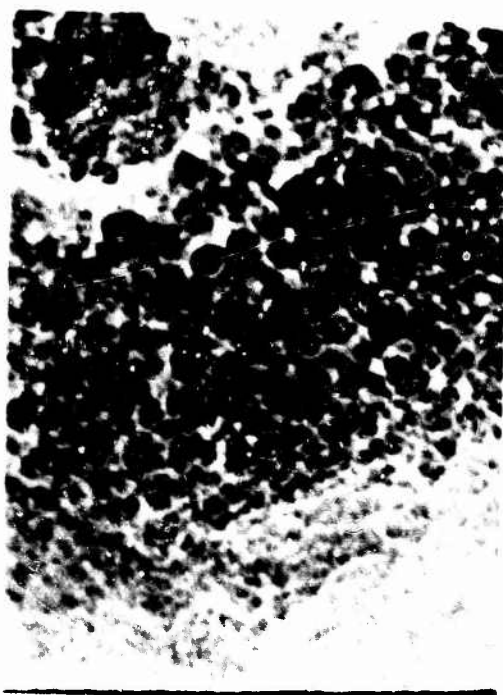
FIGURE 45e. BARE HAND CONTAMINATED CHROMIC ACID ANODIZE – SEM



800X



400X



16,000X

FIGURE 46. BARE METAL - WEDGE CRACK FAILURE BARE HAND CONTAMINATED
CHROMIC ACID ANODIZE



400X



800X



4000X



15,000X

FIGURE 47. ADHESIVE - WEDGE CRACK FAILURE BARE HAND CONTAMINATED CHROMIC ACID ANODIZE

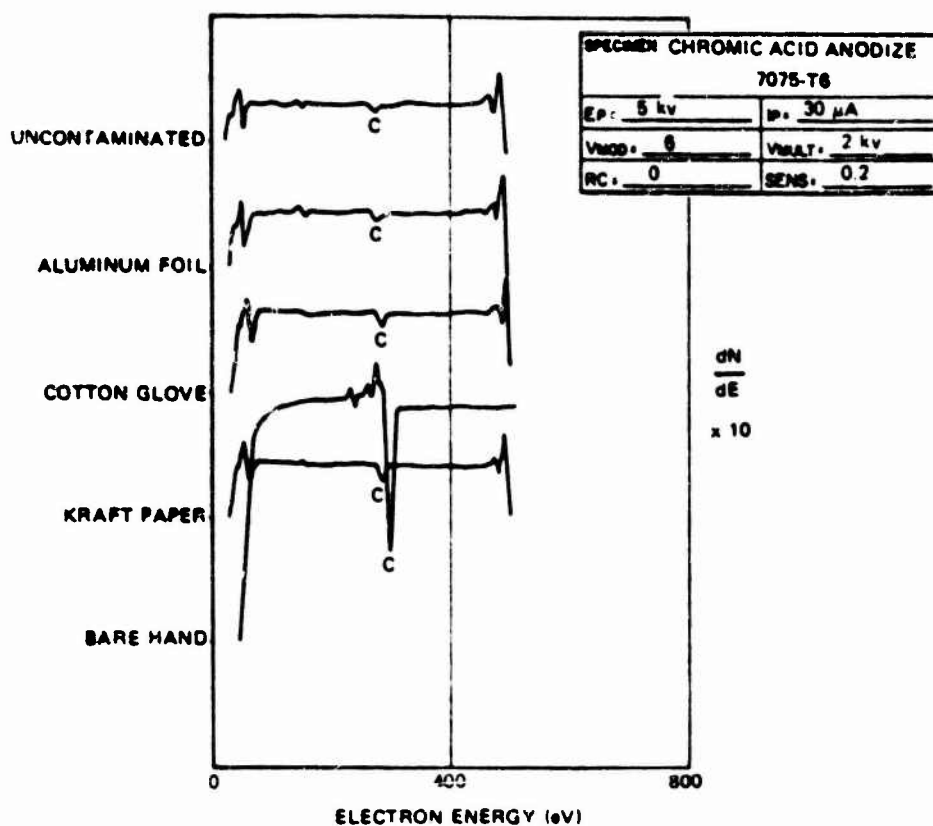


FIGURE 48. CHROMIC ACID ANODIZE - AUGER SPECTRA

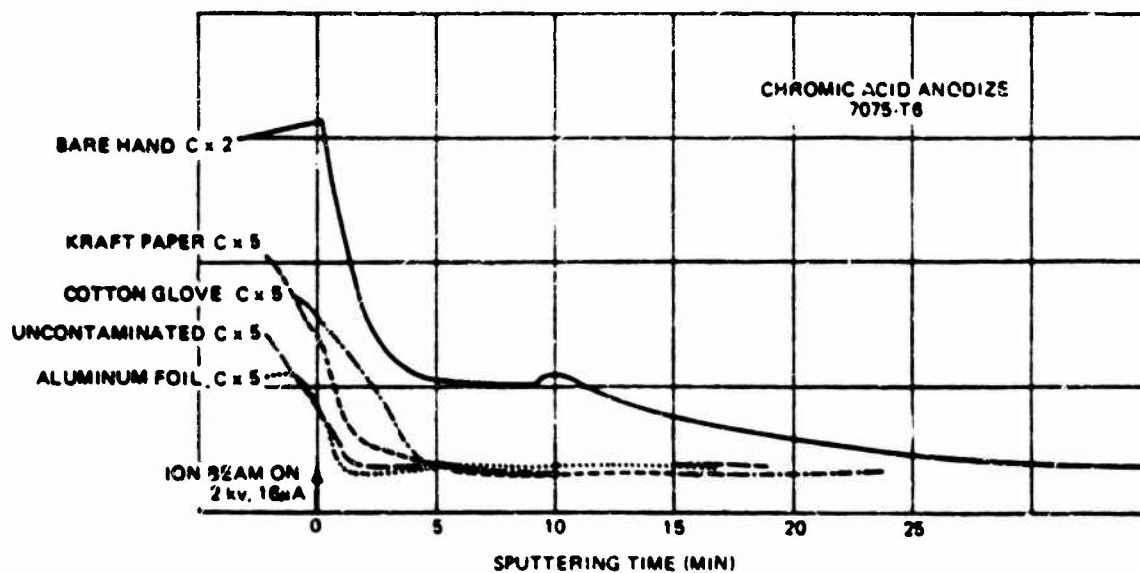


FIGURE 49. CHROMIC ACID ANODIZE - CARBON DEPTH PROFILE

4.6 Controlled Abrasion-Contamination

A Balance Beam Scrape Adhesion Tester, as described on Page 320 of the Paint Testing Manual 13th Edition 1972, ASTM Special Technical Publication 500, was modified to replace the stylus with a 0.5 inch wide, one inch diameter cylinder pivoted to allow full contact along the 0.5 inch width when used on a panel. This device was used to provide a known wiping load. The surface of the cylinder was covered by clean etched aluminum foil or kraft paper and drawn across the surface of phosphoric acid anodized wedge crack panels under a load of 1 kilogram. Three adjacent 1/2 inch wide strips were wiped on each of the test panels in the area to be exposed in the wedge crack test after bonding.

Cohesive failure was exhibited by the wedge crack test specimens when wiped with etched aluminum foil while adhesive failure was seen on the specimens wiped with kraft paper.

4.7 Fokker Contamination Tester

The Fokker Contamination Tester was used to monitor an uncontaminated phosphoric acid anodized surface and a kraft paper rubbed anodized surface. The Fokker potential of the uncontaminated specimen starts at 200 mv just after anodize and stabilizes within 48 hours at 260 to 300 mv when stored in a covered container in a laboratory atmosphere. The kraft paper contaminated phosphoric acid anodized surface starts at 500 mv just after anodize and contamination and stabilizes with 48 hours at 300 to 340 mv. When kraft paper contaminated surfaces were bonded after the 48 hour conditioning, the wedge crack failures were grossly adhesive just as the surfaces bonded immediately after contamination.

4.8 Physical and Chemical Analysis of Contamination-Abrasion

After examination of contaminated samples with ellipsometry, photo electron emersion (PEE), surface potential difference (SPD), and water contact angle, Dr. T. Smith of the Rockwell Science Center concluded that the changes noted were consistent with a physical disturbance of the anodic film.

Phosphoric acid anodized surfaces of 7075 clad have been analyzed by Auger electron spectroscopy, X-ray photoelectron spectroscopy (XPS), ion scattering spectroscopy (ISS), and by both positive and negative secondary ion mass spectroscopy (SIMS). Three surfaces were used in this study: a fresh, untouched anodized surface, an anodized surface which had been lightly rubbed with a cotton glove, and an anodized surface which had been rubbed with kraft paper.

The oxide formed by phosphoric acid anodizing has a spongy appearance at high magnification and it had been observed that by rubbing this surface the top of the spongy layer was crushed and smeared out. It had also been found that an oxide which had been rubbed with either a cotton glove or kraft paper was a poor surface for adhesive bonding. To determine whether the poor bonding was due to the deformation of the surface or to contamination transferred during the rubbing, the surfaces were examined by the techniques stated above and the surface features were photographed in a scanning electron microscope.

Figure 50 shows Auger spectra on and profiles through the oxide layers. The spectra were made before and after the argon ion beam sputtering used for eroding the surface in the profiling. The control surface (P) appears clean and shows a small carbon peak alongside a larger calcium peak. The calcium is a result of rinsing the surface with tap water. In the profiles, the peak labeled C is actually a combination of carbon and calcium, and the level maintained throughout the oxide is mainly due to calcium.

The spectrum for the cotton glove wiped oxide (PG) shows a larger carbon level than did PC. As soon as the ion bombardment began, the carbon level dropped sharply. The calcium level remained at a constant low level through the oxide. In all of these profiles, the phosphorus concentration appears to be increased by the presence of the electron beam.

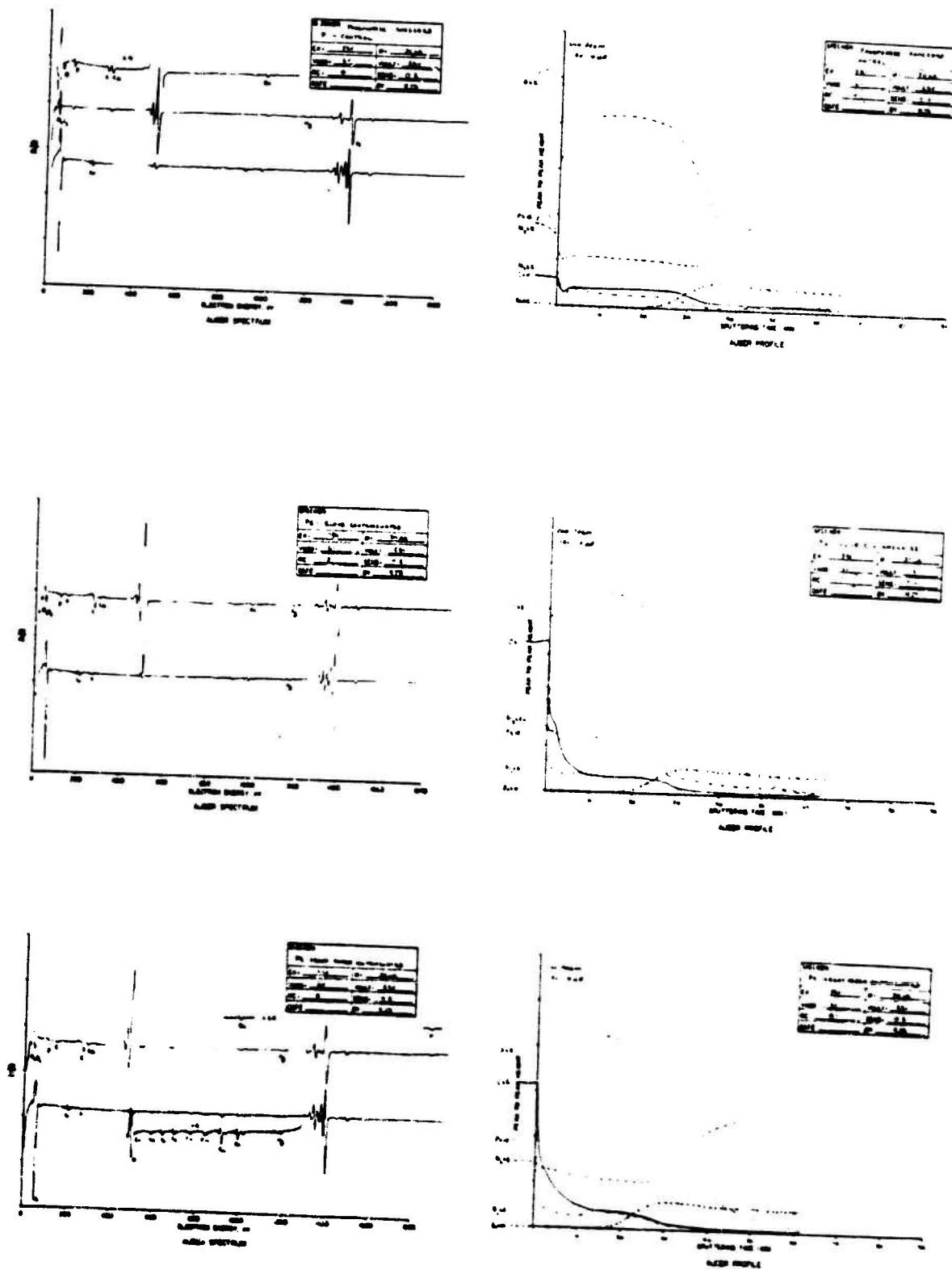


FIGURE 50. AUGER SPECTRA AND PROFILES OF CONTAMINATED PHOSPHORIC ACID ANODIZE

The surface which was rubbed with kraft paper (PK) had a higher carbon level than did P or PG. Again, the carbon profile plotted consists of both carbon and calcium as it did for P, but as PG shows, the carbon drops rapidly when struck by the ion beam but the calcium level remains at approximately the same value throughout the oxide layer. This observation has been confirmed by continuously monitoring the carbon and calcium line shapes on a strip chart recorder while the profiles are being made. An enlarged section in PK made after profiling shows the alloying elements Cu and Zn. The unlabeled lower energy peaks are probably iron and manganese.

The spectrum of the sample rubbed with kraft paper (PK) shows a small peak near 1000 ev which has been identified as zinc but could be sodium. Without more details concerning the shape of the line it is not possible to remove this ambiguity. This sample also appears to have a slightly thinner oxide layer than P or PG as determined by the time required to sputter to the crossover point of the oxygen and low energy (65 ev) aluminum peak-to-peak intensities. Also, the zinc level begins to increase at about the time the oxygen level begins to decrease, as it does in the PG profiles, but reaches a constant value somewhat before the aluminum lines flatten off. Carbon has a scale factor of X5 for PK, as compared to 100 for PG and P.

Figure 51 shows XPS measurements made with samples P, PG, and PK by Physical Electronics Industries. The carbon line designated 1-S is shown expanded on the right hand side of the figure. The intensity of the carbon photoelectrons from PG and PK is about twice that from P, but there are no large shifts in the centroids of the peaks. The asymmetrical shape of the peaks for the P and PG samples suggests the possibility of an additional low level peak on the high energy side.

These photoelectron spectra indicate that all the surfaces contain sodium, and that zinc was not detected. The spectrum for P and possibly in PG and PK identifies tin, which is not shown in the Auger analysis.

Similar XPS spectra made at the McDonnell Douglas Research Laboratories (Figure 52) show that cotton glove and kraft paper contact of the clean adherend produced a five fold increase in the surface carbon.

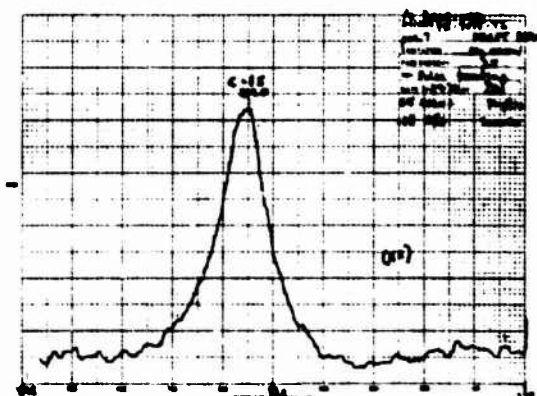
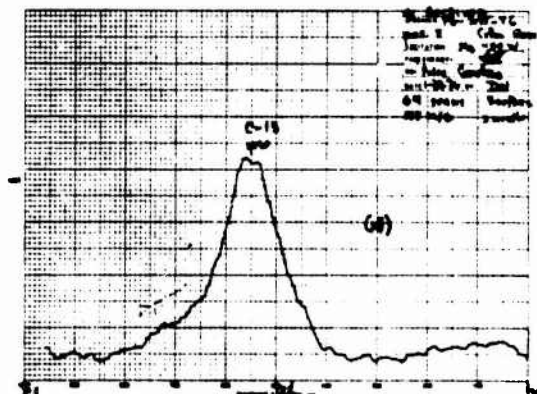
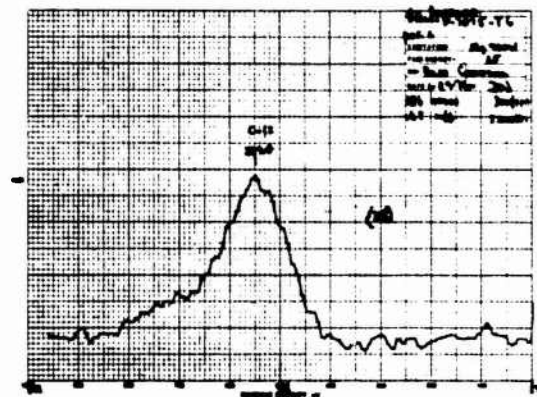
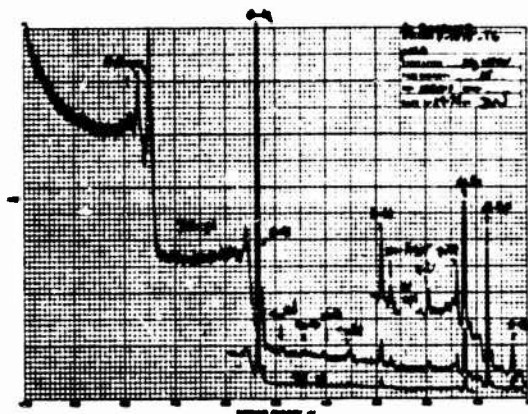


FIGURE 51. XPS ANALYSIS OF SPECIMENS P, PG AND PK

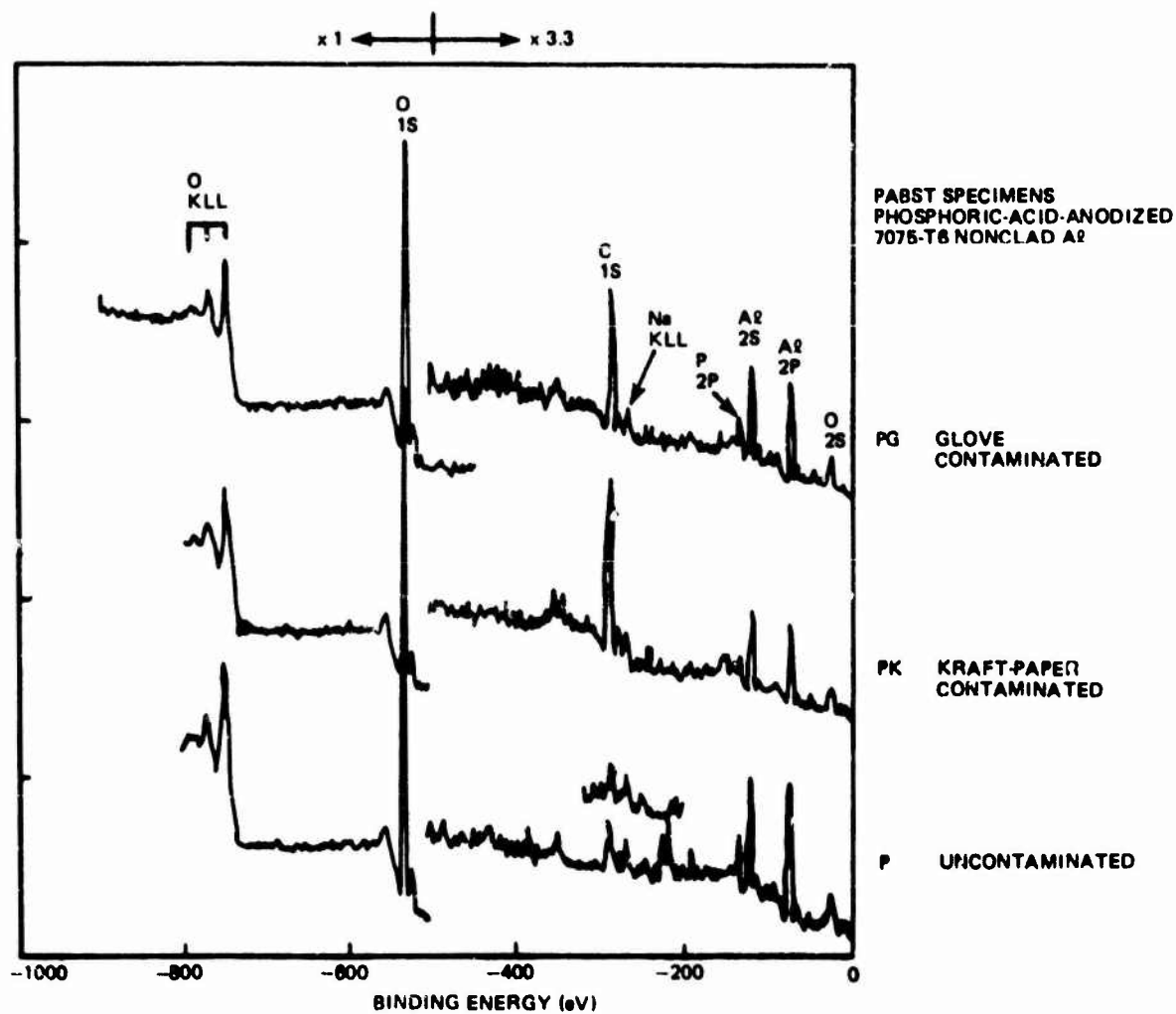


FIGURE 52. XPS ANALYSIS OF CONTAMINATED SPECIMENS

The ISS spectra for P, PG and PK and for the cotton gloves G and kraft paper K used to wipe the anodized surfaces are shown in Figure 53. The clean surface as revealed by ISS contains fluorine, potassium, bromine and lead in addition to the other elements shown in the Auger and XPS analyses, although the peak indicated as potassium could be calcium since these elements are difficult to resolve. The PG and PK samples had a greater concentration of carbon than did the untouched surface. Both had somewhat larger fluorine peaks, and lower bromine peaks.

The spectra for G and K have large carbon peaks as expected, and large silicon and potassium/calcium peaks. The kraft paper itself also contains a small amount of lead.

The positive SIMS spectra are shown in Figure 54. Those on the left hand side were obtained by 3M and those on the right hand side are from analyses made at AFML. The 3M spectra indicate that the sodium level is higher on PG and PK than on P, but this is not the case in the AFML spectra where PG has the smallest sodium peak. The AFML spectra have mass peaks at 12, 13, 14, 15, 19, and 20 corresponding to C^+ , CH^+ , N^+ , CH_3^+ , F^+ , and HF^+ , but the carbon peaks are about the same height for all samples. The potassium peak decreases between P and PG and again between PG and PK.

In the negative SIMS spectra of Figure 55, oxygen is the most prominent peak. The carbon peaks for P, PG and PK all are small and there does not seem to be an increase in height when comparing P to PG and PK. However, the C/O and CH/O ratios, after the first scans, are larger for PG and PK than they are for P. The fluorine peak in PG is larger than for P and PK and there is an indication of C_2 .

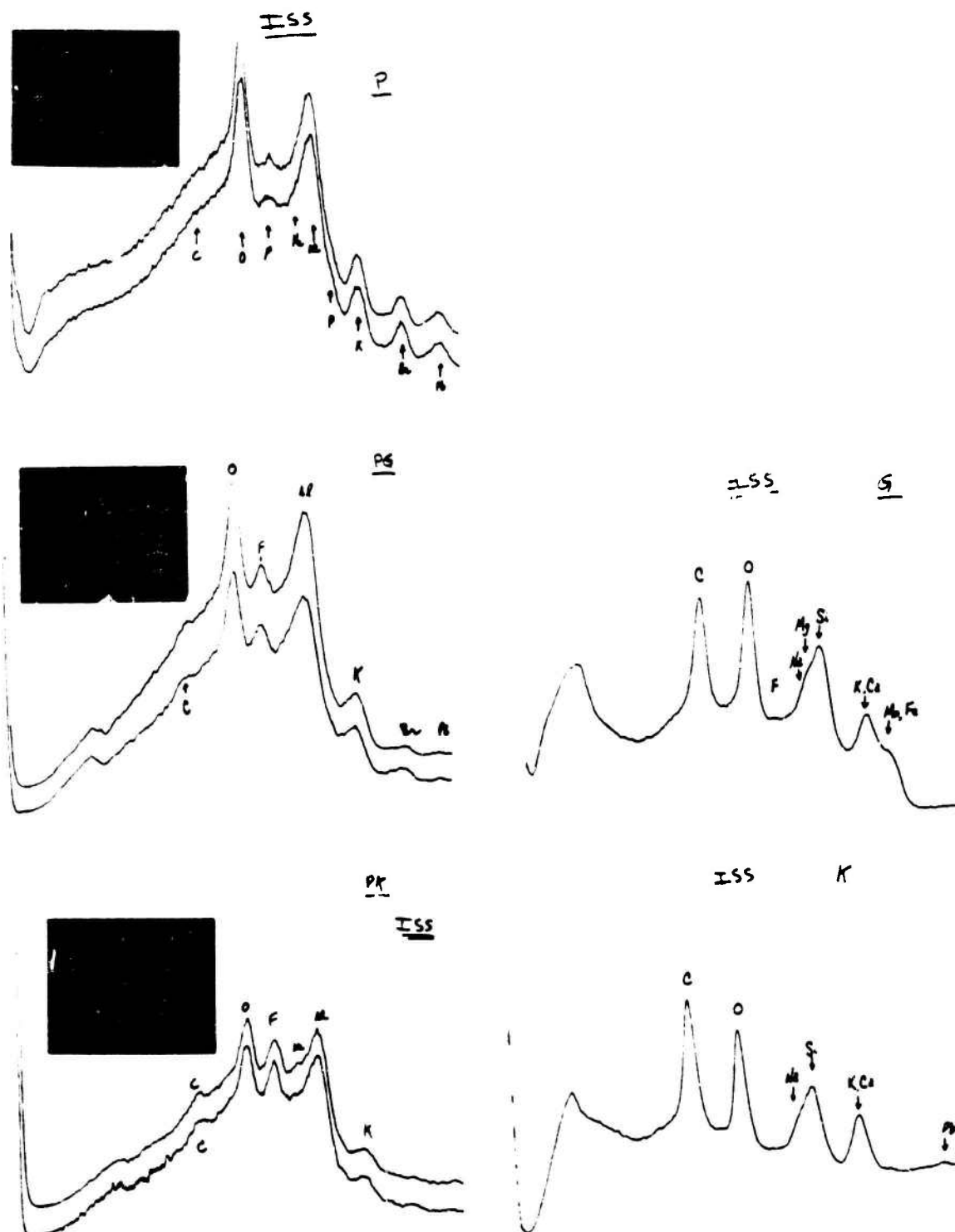


FIGURE 53. ISS ANALYSIS BY 3M OF SPECIMENS P, PG AND PK

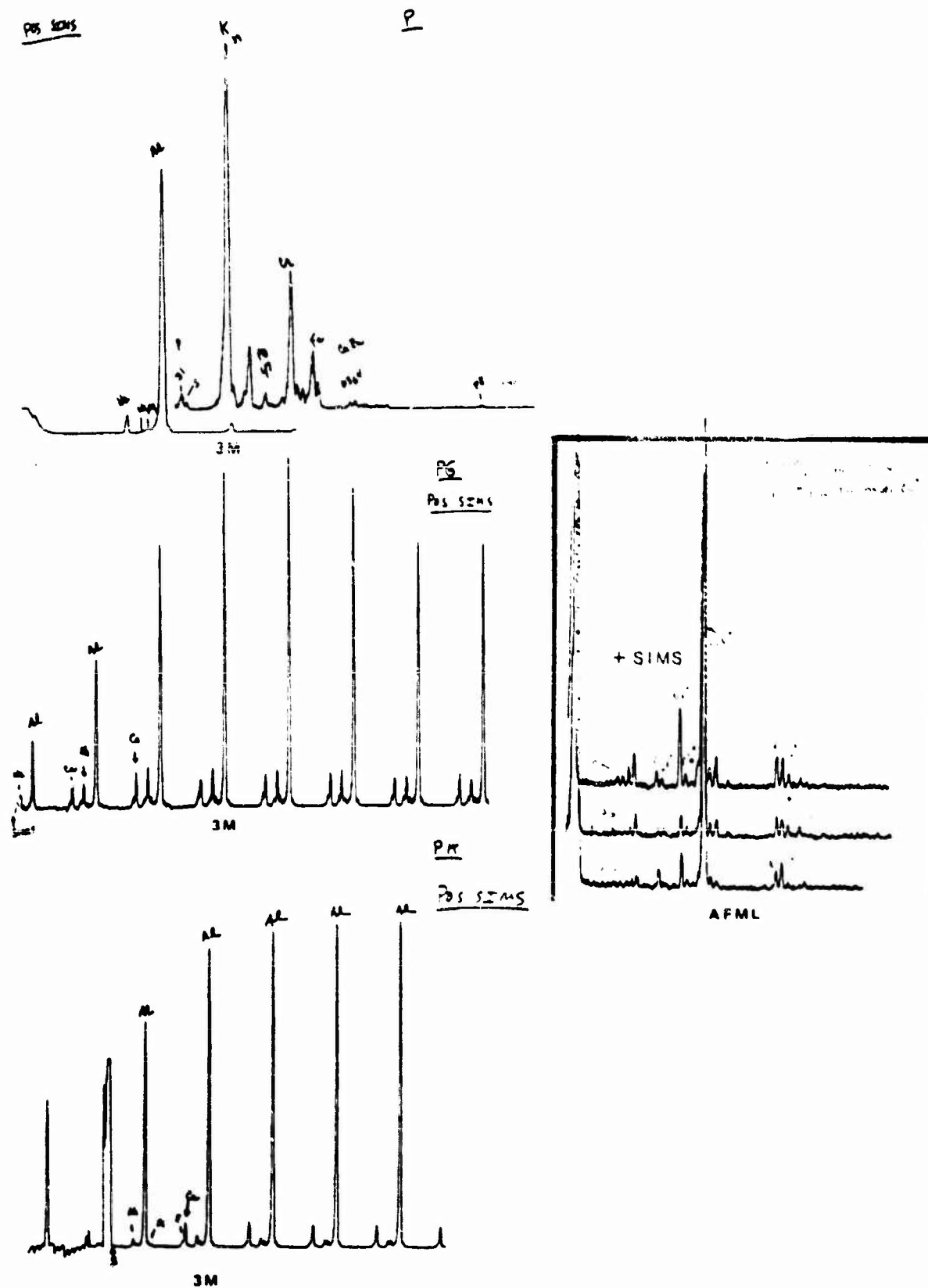


FIGURE 54. POSITIVE SIMS ANALYSIS OF CONTAMINATED SPECIMENS

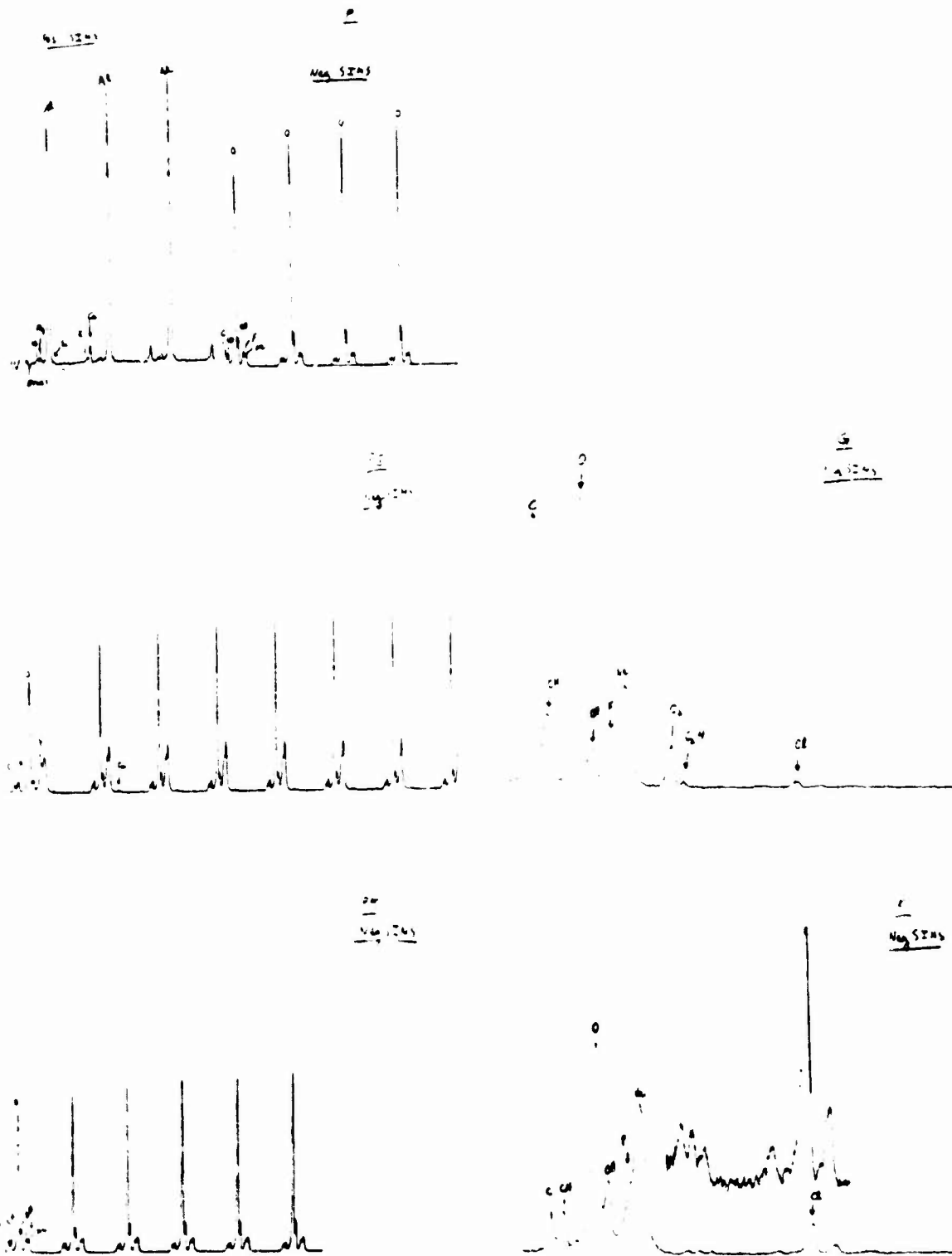


FIGURE 55. NEGATIVE SIMS ANALYSIS OF SPECIMENS P, PG AND PK

Table 10 summarizes the elements detected by the various analytical techniques. All methods except SIMS showed that there was an increased level of carbon on PG and PK as compared to specimen P. Only XPS and ISS were able to detect lead, and only ISS detected bromine, the origin of which has yet to be determined. The group of elements from mass 12 to mass 15 in the AFML SIMS spectra may reflect the composition of the background gases in the vacuum chamber rather than the surface composition itself.

The XPS of the individual carbon lines is perhaps the most powerful method of studying this form of contamination. It would be possible to mathematically resolve these lines into two or more components to chemical nature of the carbon, and this analysis would be aided by examination of the cotton glove and kraft paper themselves.

TABLE 10
SPECTRA REVEALED BY DIFFERENT METHODS OF ANALYSIS

	AUGER	XPS ¹	ISS ¹	+SIMS ¹	-SIMS ¹
Al	L	L	L	L	
AlO	-	-	-	S	
Al ₂ O ₃	L				
N	-	S	-	-	
P	M	M		S	
PO	-	-	-	S	
S	S			VS	
O	L	L	L		L
Ca	S	S	M ²		
CH					S
C	M	S	S	S	S
C ₂					
Mn	S ⁵		S ³		
Fe	S ⁵		S ³	S	
Cu	S ⁵			VS	
Zn	S			VS	
Cr	S ⁵			S	
Mg	S		S ⁴	VS	
Na	?	S	S ⁴	M	
K			M ²	M	
Sn		S			M
OH					
Br			M		
F			M	S	M
Cl					S
Pb		VS	S		
Si			M	VS	

¹ BEFORE PROFILING ONLY

² K AND Ca NOT RESOLVED

³ Mn AND Fe NOT RESOLVED

⁴ Mg AND Na NOT RESOLVED

⁵ AFTER PROFILING

L - LARGE

M - MEDIUM

S - SMALL

VS - VERY SMALL

SECTION V

CORROSION RESISTANCE AND COATINGS

5. Introduction

This section of the test program was conducted to determine the corrosion resistance of the proposed surface treatment and coatings.

By design, the alloys used in the PABST Program are nonclad. The basic corrosion protection is provided by the surface treatment, adhesive primer and the coatings. The use of the adhesive bonding surface treatment and adhesive primer over the total surfaces of all bonded details was considered to provide the best base for subsequent coatings. The durability tests in Section I proved a high level of environment resistance of the surface treatments and the adhesive primers. The cured adhesive primer also exhibited good paint base properties.

The selection of candidate coatings for protective purposes was based on historical in-service performance, maintainability, and reliability under normal and adverse aircraft environmental conditions.

Specimen configurations are shown in Figure 74. Test conditions are defined in Table 32.

5.1 Material and Processes

The adhesive primers, adhesives and coating systems used in the test program are listed. The surface treatment noted in the charts are the optimum systems as determined in Section I.

5.1.1 Coating and Adhesive Systems

The following coating and sealant combinations were evaluated. (See Table 11.)

TABLE 11
COATING SYSTEM DESIGNATION

CODE	COATING SYSTEM
C-1	MIL-P-23377
C-2	MIL-P-23377 + MIL-C-83286
C-3	MIL-P-23777 + PR1432GP + MIL-C-83019
C-4	PR1432GP + MIL-C-83019
C-5	MIL-C-8514 + MIL-P-8585 + EC 843S
C-6	PR1432GP + MIL-C-83286
C-7	MIL-S-81733 + MIL-C-83019
C-8	MIL-S-81733 + MIL-C-83286

- ° MIL-P-23377, epoxy polyamide primer, DeSoto 513-703/910-736
- ° MIL-C-83019, clear urethane copcoat, Dexter-Midland 7-C-27/10-C-81
- ° MIL-C-83286, urethane topcoat, DeSoto 821X330/910X376
- ° MIL-S-81733, chromated polysulfide sealant, Product Research PR1436G
- ° Product Research PR1432GP, chromated polysulfide spray sealant
- ° Corogard System, aluminized vinyl coat, 3M EC843S over MIL-C-8514 and MIL-P-8585 primers.

The following bonding primers and adhesives were evaluated. (See Table 12.)

TABLE 12
ADHESIVE/BONDING PRIMER SYSTEMS

MANUFACTURER	PRIMER	ADHESIVE
Hysol	EA9202	EA9628
3M	XA3950	FM55
Harmco	6740	M1133
American Cyanamid	BR127	FM73

5.1.2 Coating Application and Cure

Bonding primers were applied to a dry film thickness of 0.1 to 0.3 mils., allowed to air dry 1/2 hour at ambient conditions followed by 1 hour at 250°F. When adhesives were used in the test program, they were cured to the adhesive primer at 40 psi for 1.5 hours at 250°F. MIL-P-23377 epoxy polyamide primer was applied to a dry film thickness of 1.0 to 1.2 mils and allowed to air dry a minimum period of 1 hour before overcoating. The top coating materials were applied to the following dry film thicknesses:

- a. MIL-C-83286 white urethane - 1.8 to 2.0 mils.
- b. PR1432GP sealant - 2.5 to 4.0 mils.
- c. MIL-C-83019 clear urethane - 1.8 to 2.0 mils.
- d. MIL-S-81733 sealant (PR1436G) - 15 mils minimum.

All coating systems were air dried a minimum of 7 days prior to testing.

5.1.3 Surface Treatments

The surface treatments and their operating parameters are presented in Table 13.

TABLE 13
SURFACE TREATMENT PARAMETERS

PROCESS	VARIABLES	PROCESSING LIMITS			
		PHOSPHORIC ACID ANODIZE (BAC 5555)	CHROMIC ACID ANODIZE (MDC- PS 13201	CHROMIC ACID ANODIZE (BELL- BPS 4352, REV. G)	FPL ETCH (BAC 5514)
Anodizing	Acid Concentration	11 to 16 (opq)	6 to 8 (opq)	3 (% free acid)	
	Voltage	8 to 12	17 to 23	38 to 42	
	Temperature (°F)	65 to 90	85 to 100	92 to 95	
	Time (minutes)	20 to 25	25 to 40	30 to 35	
	Water	Deionized	Deionized	Deionized	
Sealing Solution	Chemical		Potassium Dichromate	Chromic Acid	
	Concentration		2.5 to 6 (%bw)	75 to 120 (ppm)	
	Temperature (°F)		165 to 205	180 to 185	
	Time (minutes)		8 to 17	7 to 9	
	pH		3.8 to 6	3.5 to 3.8	
Etc.	Water		Deionized	Deionized	
	Sulfuric Acid 66°BE (opq)				38.5 to 41.5
	Sodium Dichromate (opq)				4.1 to 12
	Aluminum-2024 Alloy (opq of dissolve alum.)				0.20 min.
	Temperature (°F)				150 to 160
	Time (minutes)				12 to 15

5.2 Corrosion Control

The test program for corrosion control included determining the relative resistance of each unit of the total. As the next processing step was completed, the corrosion resistance was again determined.

Reference to task numbers in the text and the tables and figures assists in identification where space limits the use of the full title.

The corrosion resistance of surface treatments are as follows:

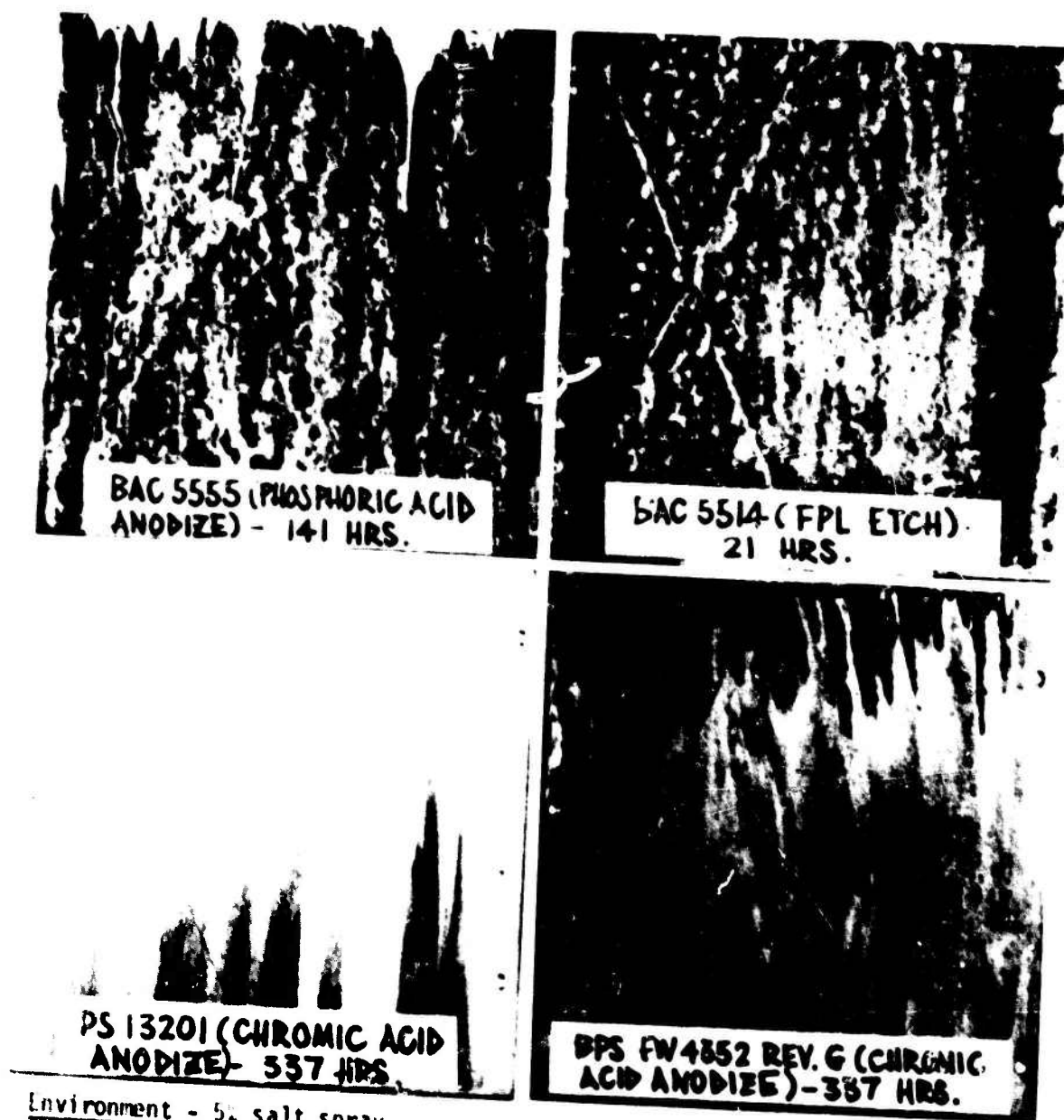
- a. The relative corrosion resistance of four aluminum surface treatments was determined in a 5% salt spray environment.
- b. Specimens were prepared and tested as noted in Table 31A, using the median range of all the processing variables. One panel from each surface treatment was coated with the PR127 primer.
- c. Data for the corrosion resistance of the primed and unprimed surface treatments are shown in Table 14. The photographs showing the comparative resistance of the unprimed panels are shown in Figure 56.

Conclusions from the tests are:

- a. Chromic acid anodize has better corrosion resistance than phosphoric acid anodize.
- b. When coated with a corrosion inhibitive bonding primer, phosphoric acid anodize is equivalent to or better than the chromic acid anodize.

TABLE 14
CORROSION RESISTANCE OF SURFACE TREATMENTS

SURFACE TREATMENT	GR127 (MILS)	SALT SPRAY TIME (HRS.)	OBSERVATIONS
Boeing BAC5514 (FPL Etch)	Unprimed	21	Heavy corrosion and pits over entire panel.
	0.13	337	Scattered blisters with corrosion underneath. No corrosion extending out from scribe.
		3000	No additional corrosion or enlargement of the blisters and corrosion.
Boeing BAC5555 (Phosphoric Acid Anodize)	Unprimed	141	Same as the unprimed FPL etch.
	0.15	3000	No corrosion extending out from the scribe mark or on the face.
MDC PS13201 (Chromic Acid Anodize)	Unprimed	337	Scattered minute corrosion pits.
	0.18	3000	Same as the primed BAC5555.
Bell Helicopter BPS FW4352 Rev. G. (Chromic Acid Anodize)	Unprimed	337	Same as the unprimed PS13201.
	0.35	3000	Same as the primed BAC5555.



Environment - 5% salt spray
 Surface Treatments (unprimed)

- Boeing - BAC5555
- Boeing - BAC5514
- MDC - PS13201
- Bell helicopter- BPS FW4352, Rev. G

FIGURE 56. SALT SPRAY EXPOSURE OF SURFACE TREATMENTS

5.2.1 Corrosion Resistance of the MDC Chromic Acid Anodize Sealing Matrix (Task 105)

The sealing parameters that provide the best corrosion resistance to chromic acid anodize were determined in a 5% salt spray environment. Specimens were prepared and tested as noted in Table 31A. One half of each specimen was coated with BR127 primer.

Results are as follows:

Data for the corrosion resistance are shown in Table 15 and in the following photographs.

Figures 57 and 58 (2024-T3 deionized water)

Figures 59 and 60 (7075-T6 deionized water)

Figures 61 and 62 (2024-T3 tap water)

Figures 63 and 64 (7075-T6 deionized water)

The sealing parameters that provide the best corrosion resistance to is obtained at the upper limits of the dichromate concentration, temperature and time using deionized water. In addition these panels exhibited minimal or no staining of the anodic surface. See Figures 58 and 60.

The BR127 primed portion of all specimens did not exhibit any coating failures or corrosion.

TABLE 15
CORROSION RESISTANCE (5% Salt Spray) OF
SEALING MATRIX SPECIMENS

		DEIONIZED				TAP			
		C ₁		C ₂		C ₁		C ₂	
		t ₁	t ₂	t ₁	t ₂	t ₁	t ₂	t ₁	t ₂
2024	T ₁	288	288	168	168	168	168	168	168
	T ₂	288	336	840	►	168	384	288	288
7075	T ₁	840	840	384	648	384	336	648	648
	T ₂	840	1000	►	►	384	►	840	840

NOTES: (1) Sealing Parameters

C = Conc. of Potassium Dichromate (% bw)

C₁ = 2.5 , C₂ = 6.0

T = Temperature (°F)

T₁ = 185 , T₂ = 205





t = time (minutes)

t₁ = 8 , t₂ = 17

pH = 3.8



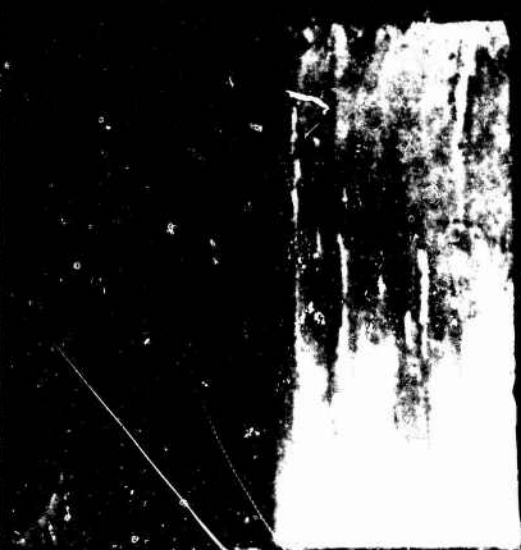

(2) Failure is as defined per MIL-A-8625. All specimens were examined at 168, 288, 336, 384, 648, 840 and 1000 hours. Hours to failure data is intended to be comparative at these periods.

► No failure at 1000 hours.

WATER	DEIONIZED	
K ₂ Cr ₂ O ₇ CONC.	2.5 % BY WEIGHT	
TIME TEMP.	8 MINUTES	17 MINUTES
185°F		
205°F		
	BR 127 UNPRIMED	BR 127 UNPRIMED





NON CLAD ALUMINUM ALLOY - 2024-T3

FIGURE 57. SALT SPRAY - CHROMIC ACID ANODIZE

WATER	DEIONIZED	
K ₂ Cr ₂ O ₇ CONC.	6.0 % BY WEIGHT	
TIME TEMP.	8 MINUTES	17 MINUTES
185°F		
205°F		
	BR 127 UNPRIMED	BR 127 UNPRIMED


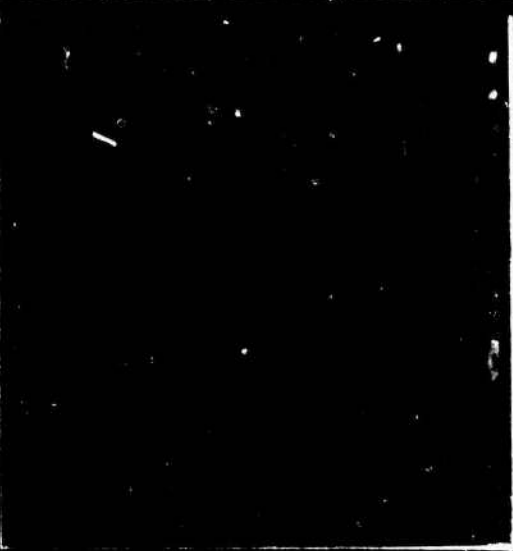


NONCLAD ALUMINUM ALLOY - 2024-T3

FIGURE 58. SALT SPRAY - CHROMIC ACID ANODIZE

WATER	DEIONIZED			
K ₂ Cr ₂ O ₇ CONC.	2.5 % BY WEIGHT			
TIME TEMP.	8 MINUTES		17 MINUTES	
185°F				
205°F				
	BR 127	UNPRIMED	BR 127	UNPRIMED


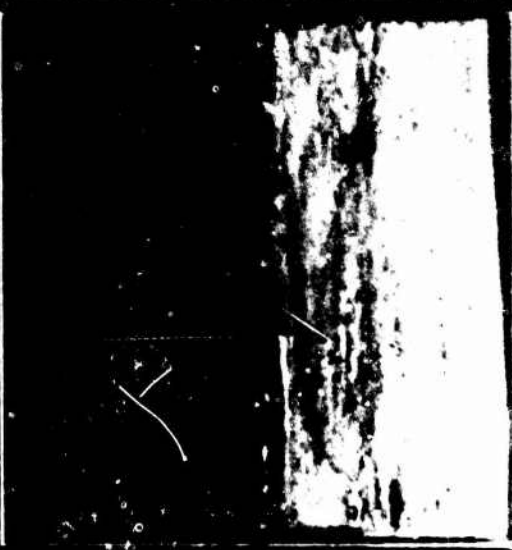


NON CLAD ALUMINUM ALLOY - 7075-T6

FIGURE 59. SALT SPRAY - CHROMIC ACID ANODIZE

WATER	DEIONIZED			
K ₂ Cr ₂ O ₇ CONC.	6.0 % BY WEIGHT			
TIME TEMP.	8 MINUTES		17 MINUTES	
185°F				
205°F				
	BR 127	UNPRIMED	BR 127	UNPRIMED





NONCLAD ALUMINUM ALLOY - 7075-T6

FIGURE 60. SALT SPRAY - CHROMIC ACID ANODIZE

WATER	TAP	
K ₂ Cr ₂ O ₇ CONC.	2.5 % BY WEIGHT	
TIME TEMP.	8 MINUTES	17 MINUTES
185°F		
205°F		
	BR 127 UNPRIMED	BR 127 UNPRIMED





NONCLAD ALUMINUM ALLOY - 2024-T3

FIGURE 61. SALT SPRAY - CHROMIC ACID ANODIZE

WATER	TAP	
K ₂ Cr ₂ O ₇ CONC.	6.0 % BY WEIGHT	
TEMP. \ TIME	8 MINUTES	17 MINUTES
185°F		
205°F		
	BR 127 UNPRIMED	BR 127 UNPRIMED


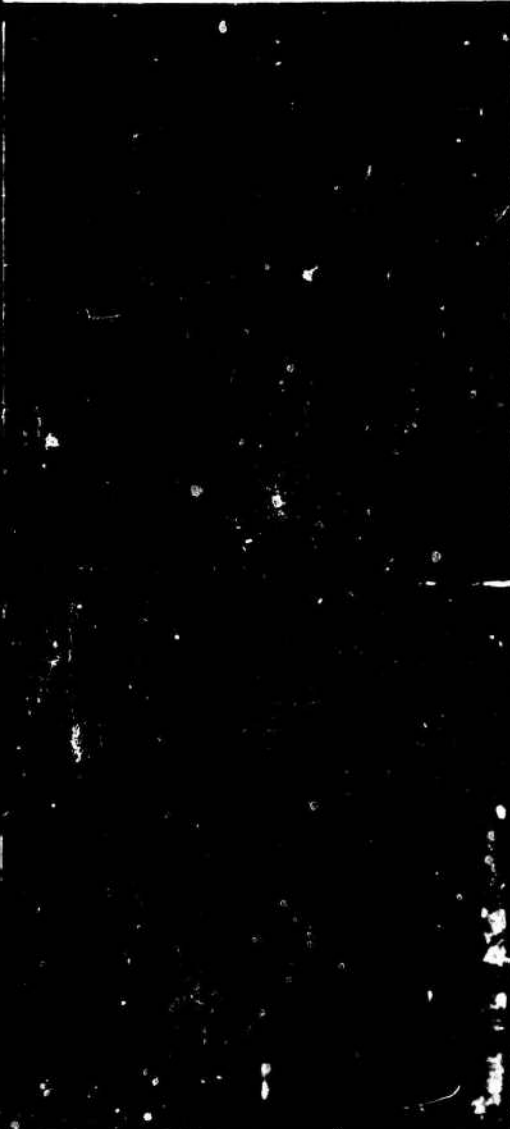
NONCLAD ALUMINUM ALLOY - 2024-T3

FIGURE 62. SALT SPRAY - CHROMIC ACID AND RIZE

WATER	TAP			
K ₂ Cr ₂ O ₇ CONC.	2.5 % BY WEIGHT			
TIME TEMP.	8 MINUTES		17 MINUTES	
185°F				
205°F				
	BR 127	UNPRIMED	BR 127	UNPRIMED

NONCLAD ALUMINUM ALLOY - 7075-T6

FIGURE 63. SALT SPRAY - CHROMIC ACID ANODIZE

WATER	TAP	
K ₂ Cr ₂ O ₇ CONC.	6.0 % BY WEIGHT	
TIME TEMP.	8 MINUTES	17 MINUTES
185°F		
205°F		
	BR 127 UNPRIMED	BR 127 UNPRIMED

NONCLAD ALUMINUM ALLOY - 7075-T6

FIGURE 64. SALT SPRAY -- CHROMIC ACID ANODIZE

5.2.2 Comparison of Bonding Primers

The environmental resistance of the four different bonding primer systems per Table 12, with and without various coating systems, was determined. Specimens were prepared and tested as noted in Table 31B.

Results are as follows:

- a. Data for the adhesion, humidity, salt spray, immersion corrosion and bilge fluid tests are shown in Table 16.

No corrosion failures were exhibited in either the salt spray humidity, bilge fluid or immersion corrosion tests. The only type of failure that occurred on any of the coating systems was adhesion. Other than the cohesive failure of the MIL-P-83286 topcoat on the 3M XA3950/FM55 system the failures were limited to system C-3 and C-4. Adhesion failures of these systems were the MIL-C-83019 clear to the PR1432GP sealant; and, the PR1432GP to the MIL-P-23377 primer. In addition, adhesion failure of the PR1432GP sealant to both the FM73 and EA9628 adhesives occurred with system C-4. This indicates that a primer is required over the adhesive flash to insure adequate adhesion.

TABLE 16
ENVIRONMENTAL TESTS - COATINGS/BONDING MATERIALS

TEST	BOND PRIMER/ ADHESIVE ①	COATING SYSTEMS OVER BOND PRIMER					
		NONE	C-1	C-2	C-3	C-4	C-5
Adhesion	BR127/FM73	P	P	P	F(2)	P	P
	EA9202/EA9628	P	P	P	P	P	P
	XA3950/FM55	P	P	F(3)	P	P	P
	6740/M1133	P	P	P	F(2)(4)	P	P
Humidity (30 days)	BR127/FM73	P	P	P	F(2)	F(2)	P
	EA9202/EA9628	P	P	P	P	P	P
	XA3950/FM55	P	P	P	F(2)(4)	F(2)	P
	6740/M1133	P	P	P	F(2)	F(2)	P
Salt Spray (2000 hrs.)	BR127/FM73	P	P	P	P	P	P
	EA9202/EA9628	P	P	P	P	P	P
	XA3950/FM55	P	P	P	P	P	P
	6740/M1133	P	P	P	P	P	P
Immersion Corrosion (30 days)	BR127/FM73	P	F	P	F(2)(4)	F(2)(5)	P
	EA9202/EA9628	P	P	P	F(2)	F(2)(5)	P
	XA3950/FM55	P	P	F(3)(6)	F(2)	F(2)	P
	6740/M1133	P	P	P	F(2)	F(2)	P
Bilge Fluid (30 days)	BR127/FM73	P	P	X	F(4)	F(2)(4)	X
	EA9202/EA9628	P	P		F(4)	F(2)	
	XA3950/FM55	P	P		F(2)	F(2)	
	6740/M1133	P	P		F(2)	P	

P = Pass F = Fail

- ① All bond systems applied over phosphoric anodized 7075-T6 nonclad aluminum. Specimen Figure 19-4.
- ② Adhesive failure of MIL-C-83019 clear to PR1432GP sealant.
- ③ Cohesive failure of MIL-P-23377 primer.
- ④ Adhesive failure of PR1432GP sealant to MIL-P-23377 primer.
- ⑤ Adhesive failure of PR1432GP sealant to adhesive.
- ⑥ Blisters per ASTM D714. Size #8 scattered randomly.

- b. Data for the exfoliation corrosion resistance are shown in Table 17 and in photograph Figure 65.

A comparison of the four bonding primers in the exfoliation corrosion environment showed the following:

- ° All of the bonding primers by themselves exhibited substantial film failures and did not inhibit corrosion, especially in the nonfastener surfaces. Of the primers, the Narmco 6740 had gross corrosion and exfoliation over the entire test surface. Overcoating each of the bond primers with MIL-P-23377 primer greatly reduced coating failure and subsequent corrosion.
- ° Wet installation of fasteners reduced the degree of coating failure around dissimilar metal areas.
- ° The overall performance of the BR127 primer under the various coating systems was better than that of the other three primers. This combination has less coating degradation around fasteners and along the cut edges. The performance of the other primers were about comparable to each other.
- ° Examination of the bond line after separating the assemblies showed:
 - (1) Mode of failure of the four different adhesives was cohesive.
 - (2) No corrosion was present in the fastener hole areas of any of the bonding systems.
 - (3) A slight amount of corrosion undercutting was found along the glue line of the Hysol 9202 and the Narmco 6740 assemblies.

TABLE 17
ACIDIFIED SALT SPRAY (4 wks) - DIFFERENT BONDING PRIMER SYSTEMS

BOND PRIMER	COAT. SYSTEM OVER PRIMER	PHOSPHORIC ANODIZED NONCLAD 7075-T6		
		FIRST SIGN (wks) OF GEN. SURF. CORR.	DISSIMILAR METAL	CUT EDGES
BR127	None	1	No Exfol.	No Exfol.
	C-1	↓	↓	↓
	C-2			
	C-3		Exfol.	
	C-4	↓	No Exfol.	
	C-5	2		
EA9202	None	1		
	C-1	↓		
	C-2			
	C-3			
	C-4	↓	↓	↓
	C-5	2	Exfol.	Exfol.
XA3950	None	1	No Exfol.	No Exfol.
	C-1	↓	↓	↓
	C-2			
	C-3			
	C-4			
	C-5	↓	↓	↓
6740	None	1	Exfol.	Exfol.
	C-1	↓	No Exfol.	No Exfol.
	C-2			Exfol.
	C-3			No Exfol.
	C-4			Exfol.
	C-5	↓	↓	↓

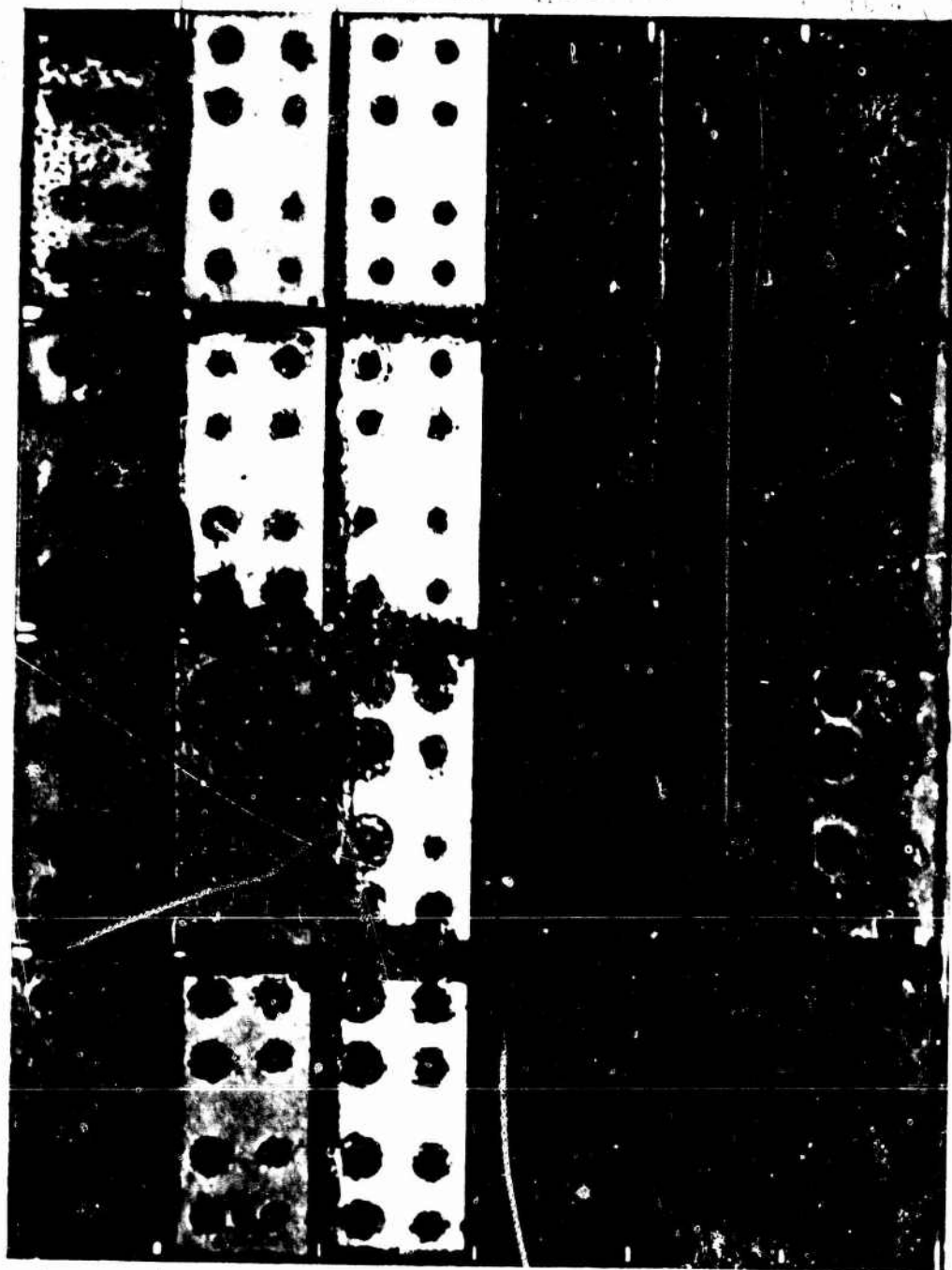


FIGURE 65. ACIDIFIED SALT SPRAY - COMPARISON OF PRIMERS
OVER PHOSPHORIC ACID ANODIZE

- c. Data for the beach exposure are shown in Table 18 and in photograph Figure 66.

Dissimilar metal beach panels of the top coated bonding primers all exhibited corrosion of varying degrees. After 18 months exposure, the BR127 panels had minimal corrosion around the fastener areas and was the only primer that did not have any evidence of exfoliation corrosion.

TABLE 18
BEACH EXPOSURE OF BONDING PRIMERS

FINISH SYSTEM OVER BOND PRIMER	BONDING PRIMER			
	HYSOL 9202	3M 3950	NARIMO 6470	AMER. CY. BR127
MIL-P-23377 & MIL-C-83286	E	E	E	C
Corogard	E	E	E	C

NOTE: Corrosion on panels is in dissimilar metal areas.

E = Exfoliation

C = Pitting Corrosion

Exposure Period = 18 months









BONDING PRIMER	FINISH SYSTEM OVER BONDING PRIMER	
	COROGARD	MIL P 23377 + MIL C-83286
HYSOL 9202		
3M 3950		
NARMCO 6470		
AMER CYMD BR127		

FIGURE 66. BEACH EXPOSURE - PRIMER COMPARISON

5.2.3 Surface Treatment Protection

The ability of the various coating systems to protect the phosphoric acid or chromic acid anodize surfaces was determined. Specimens were prepared and tested as noted in Table 31C. The only bond primer used was BR127. In order to observe the exfoliation corrosion resistance of finish systems in a nondissimilar metal condition, additional panels were prepared without any fasteners and only scribed through the coating. Non-clad 2024-T3 and 7075-T6 aluminum alloy panels were chromic and phosphoric acid anodized, primed with BR127 and over coated with the MIL-P-23377 primer and MIL-C-83286 topcoat system (C-2). In addition the following two panels were made for comparison:

- ° A 7075-T6 phosphoric acid anodize panel with BR127 was overcoated with the MIL-P-23377 primer plus PR1432GP sealant plus MIL-C-83019 topcoat system (C-3).
- ° A 7075-T6 phosphoric acid anodize panel with Hysol EA9202 adhesive primer was overcoated with system C-2.

The results of the surface treatment protection tests are as follows:

Data for the adhesion, humidity, salt spray, immersion corrosion and bilge fluid are shown in Table 19.

In the adhesion (wet) test, the only failure was exhibited by system C-2 (MIL-P-23377 primer overcoated with the MIL-C-83286 topcoat). The cohesive failure of the primer has been experienced from time to time in laboratory tests, however no such failures have been reported in service.

In the humidity test, none of the coating systems exhibited any film failures such as blistering, cracking or loss of adhesion after 30 days at 140°F and 100% relative humidity.

For the 5% salt spray test, all of the coating systems provided corrosion protection without any coating failures. However, systems C-3 and C-4 had a tendency to exhibit very slight adhesive failure of the clear (MIL-C-83019) to the PR1432GP sealant at the intersection of the scribe line.

TABLE 19
ENVIRONMENTAL TESTS - COATED ANODIC SURFACES

TEST	TYPE ANODIZE ①	COATING SYSTEMS OVER BR127 PRIMER					
		CONTROL (NONE)	C-1	C-2	C-3	C-4	C-5
Adhesion	Phosphoric	P	P	F ②	P	P	P
	Chromic	P	P	P	P	P	P
Humidity (30 days)	Phosphoric	P	P	P	P	P	P
	Chromic	P	P	P	P	P	P
Salt Spray (2000 hrs)	Phosphoric	P	P	P	P	P	P
	Chromic	P	P	P	P	P	P
Immersion Corrosion (30 days)	Phosphoric	P	P	P	F ③④	F ③	P
	Chromic	P	P	X	F ③④	F ③	X
Bilge Fluid (30 days)	Phosphoric	P	P		F ④	F ③④	
	Chromic	P	P		F ④	F ③	

P = Pass F = Fail

① Anodized nonclad aluminum alloy - 2024 and 7075. Specimen Figure 19-1.

② Cohesive failure of MIL-P-23377 primer.

③ Adhesive failure of MIL-C-83019 clear to PR1432GP sealant.

④ Adhesive failure of PR1432GP sealant to MIL-P-23377 primer.

Data for the exfoliation corrosion resistance of the dissimilar metal panels are shown in Table 20 and in photograph Figures 67 and 68.

In the acidified salt spray test, the following decreasing order of corrosion resistance of the various aluminum alloys and tempers were noted for both the chromic and phosphoric acid anodize assemblies.

<u>General Surface Corrosion</u>		<u>Exfoliation Corrosion</u>
2024-T3	DECREASING	7075-T73 and T76
7075-T73		2024-T3
7075-T6		7075-T6
7075-T76		

- ° Phosphoric acid anodize exhibits equal or better resistance to exfoliation corrosion better than does chromic acid anodize.
- ° The 7075-T73 and T76 tempers show no exfoliation with either anodize. The 2024-T3 exhibits exfoliation only with the chromic acid anodize and where the fasteners are installed dry. The 7075-T6 exhibits the least resistance to exfoliation whether it is anodized in chromic or in phosphoric. Dry or wet installation of the fasteners does not make any difference with this alloy.

Inspection of the bond line after the assemblies were mechanically separated with a metal wedge showed the following:

- ° Mode of failure of the FM73 adhesive was all cohesive.
- ° No corrosion of any type was present in the fastener hole area of the bond line on any of the panels.
- ° No corrosion had extended into the bond line along the cut edges of the 7075-T6 and 7075-T73 assemblies. A slight amount of corrosion undercutting had occurred along the glue line at the edges or corners on some of the 2024-T3 and 7075-T76 assemblies. In all cases corrosion attack was minimal in the glue line.

TABLE 20
ACIDIFIED SALT SPRAY - COATED ANODIC SURFACES

NONCLAD ALUM. & TEMPER.	COAT SYSTEM (OVER BRI27)	PHOSPHORIC ANODIZE			CHRONIC ANODIZE		
		FIRST SIGN (HKS) OF GENERAL SURF. CORR.	DISSIMILAR METAL	CUT EDGES	FIRST SIGN (HKS) OF GENERAL SURF. CORR.	DISSIMILAR METAL	CUT EDGES
2024-T3	None	2	No Exfol.	No Exfol.	3	Exfol.	No Exfol.
	C-1	3				No Exfol.	
	C-2						
	C-3						
	C-4				1	Exfol.	
	C-5				3	No Exfol.	
7075-T6	None	1			1	Exfol.	Exfol.
	C-1					No Exfol.	
	C-2						
	C-3		Exfol.			Exfol.	No Exfol.
	C-4		No Exfol.			No Exfol.	Exfol.
	C-5	2			3	Exfol.	
7075- T73	C-1				1	No Exfol.	No Exfol.
	C-2						
	C-3						
	C-4						
	C-5				2		
7075- T76	C-1				1		
	C-2						
	C-3						
	C-4						
	C-5				2		

Exposure Period = 4 Weeks

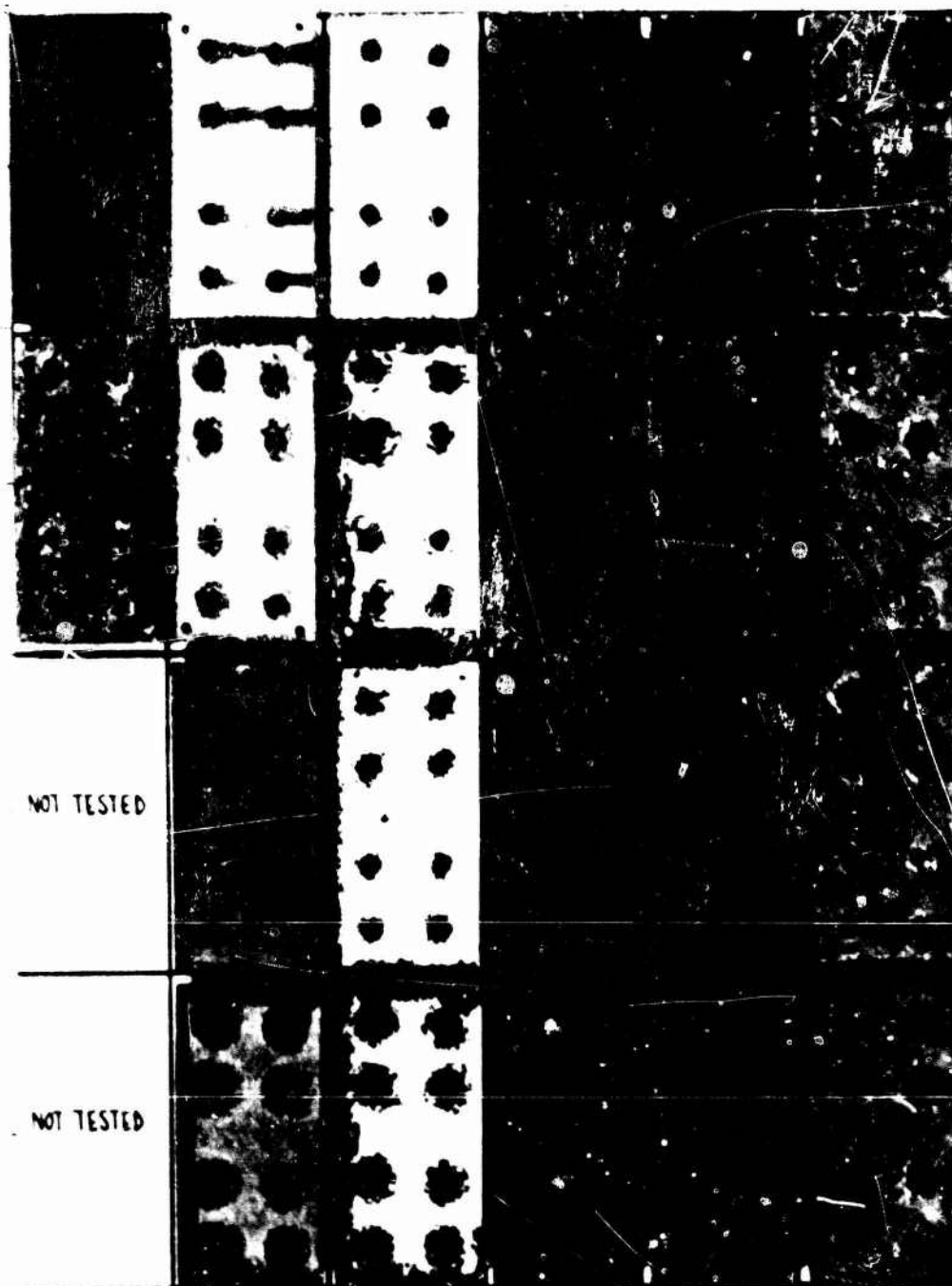


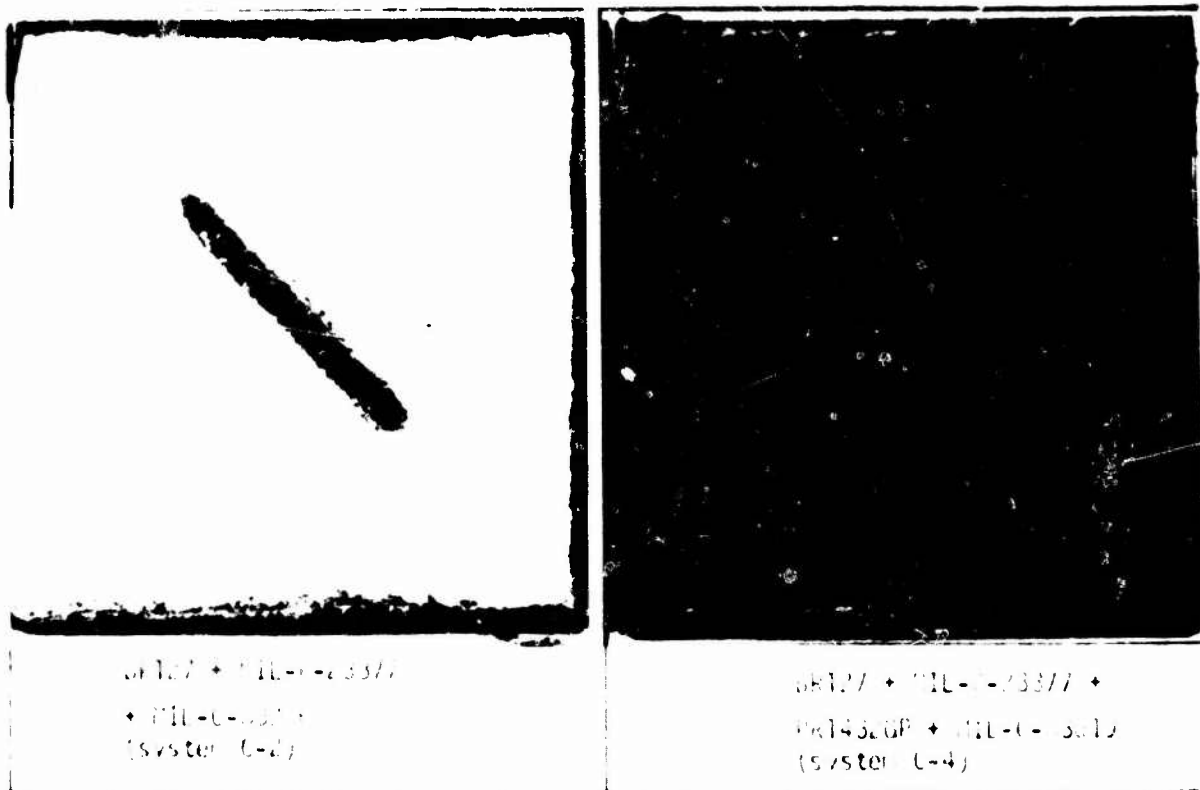
FIGURE 68. ACIDIFIED SALT SPRAY - COATED CHROMIC ACID ANODIZED ALUMINUM

Performance of the protective coating systems were noted as follows:

- Coating failures such as blistering and lifting occurring around the fastener heads were of a lesser magnitude on the phosphoric anodize as compared to the chromic anodize. Data shows that the amount and degree of film failure of any of the coating system is mainly a function of the corrosion resistance of the aluminum alloy, the temper and the anodic process. This can be seen in photographs Figures 66 and 67.
- Wet installation of fasteners using MIL-S-18733 sealant helps reduce the degree of coating failure in dissimilar metal contact areas.
- BR127 primed only panels require a coat of MIL-P-23377 (system C-1) to adequately resist corrosion in nonfastener areas.
- System C-2 (MIL-P-23377 and MIL-C-83286) and system C-5 (Corogard) can be considered comparable. However, C-5 did not inhibit exfoliation on the fastener area of the chromic acid anodized 7075-T6 assembly.
- System C-3 and C-4 are comparable. The use of the MIL-P-23377 primer under the PR1432GP in C-3 did not appear to improve the exfoliation resistance.

Exfoliation corrosion resistance data of nondissimilar metal panels are shown in photograph Figures 69, 70 and 71.

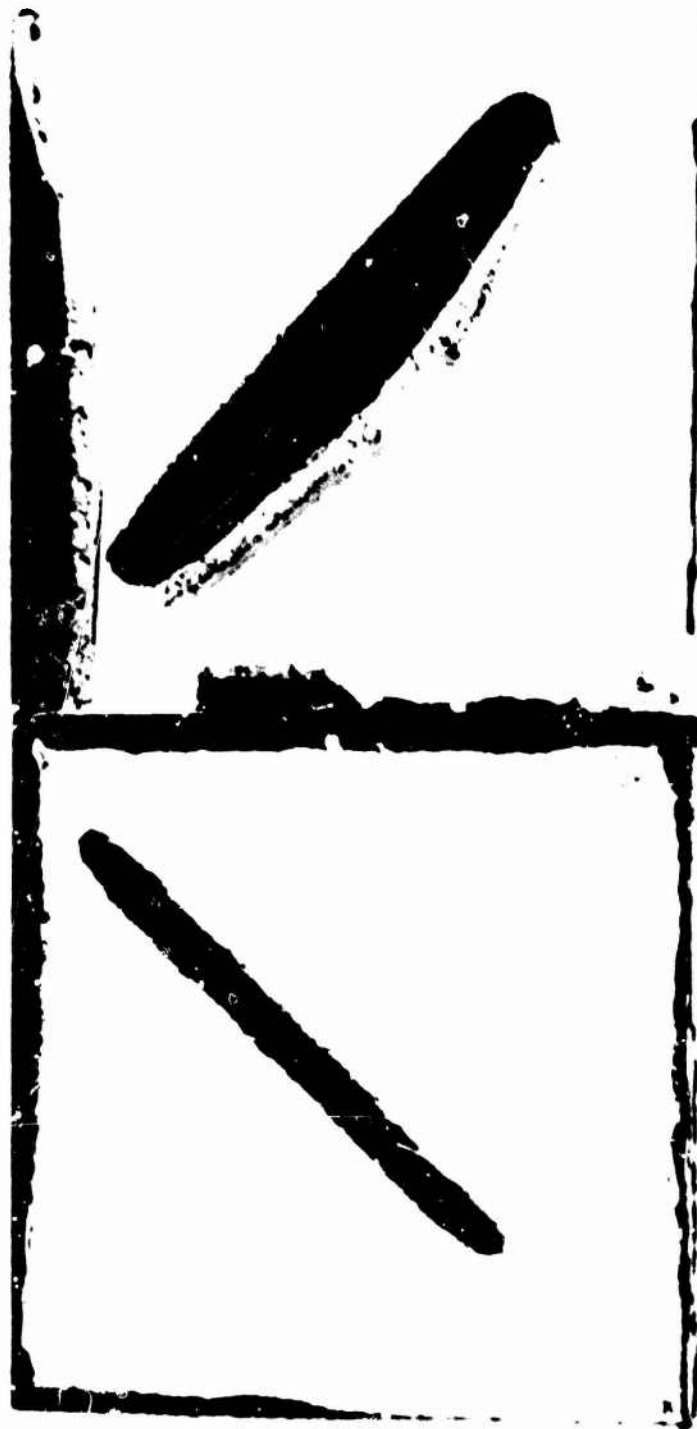
- A dissimilar metal condition is necessary to obtain accelerated corrosion data.
- The 2024-T3 alloy shows better resistance to exfoliation corrosion than the 7075-T6 alloy for both chromic and phosphoric anodizes.
- There is only a small degree of difference between the resistance of systems C-2 and C-3.
- BR127 primer provides better exfoliation corrosion resistance than EA9202 as previously noted.
- Although corrosion took longer to occur, the results correlated with the data from exfoliation tests previously run with dissimilar metal.



Substrate: Inconel 718-Ti with Phosphoric Acid Anodize

Exposure time acidified salt spray = 2000 hours

FIGURE 69. ACIDIFIED SALT SPRAY — COMPARISON OF COATING SYSTEMS



BOND PRIMER:

BR 127

EA 9202




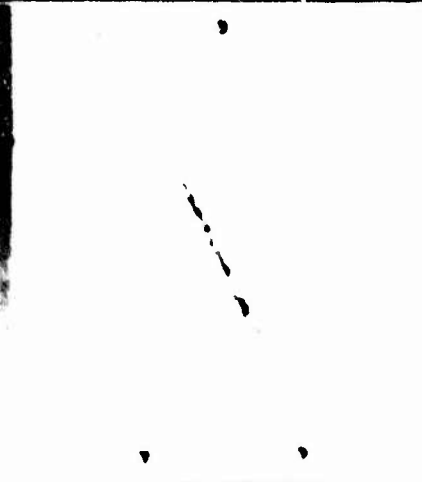
COAT. SYSTEM:

MIL-P-23377 + MIL-C-83286

SUBSTRATE:

NONCLAD 7075-T6 + PHOSPHORIC ACID ANODIZE

FIGURE 70. ACIDIFIED SALT SPRAY - COMPARISON OF ADHESIVE PRIMERS

ALUM ANODIZE	NONCLAD 7075-T6	NONCLAD 2024-T3
PHOSPHORIC		
CHROMIC		

COATING SYSTEM - BR127 + MIL-P-23377 + MIL-P-83286

Exposure Period - 2000 hours

FIGURE 71. ACIDIFIED SALT SPRAY ANODIZE AND ALUMINUM ALLOYS

Data of the beach exposure are shown in Table 21. The corrosion resistance of the topcoated dissimilar metal specimens after 21 months of beach exposure indicate a correlation in the corrosion results with the accelerated laboratory tests (exfoliation corrosion).

LAB ACIDIFIED SALT SPRAY (1 Mo)		BEACH EXPOSURE (21 mos)
7075-T73	Decreasing Order Corrosion Resistance ↓	7075-T73
7075-T76		7075-T76
2024-T3		2024-T3
7075-T6		7075-T6

TABLE 21
BEACH EXPOSURE

SURFACE TREATMENT	FINISH OVER BR127	ALUM. ALLOY (NONCLAD)			
		2024	7075		
		T3	T6	T73	T76
Phosphoric Acid Anodize	MIL-P-23377 & MIL-C-83286	C	C	N	C
	Corogard	E	E	N	C
Chromic Acid Anodize	MIL-P-23377 & MIL-C-83286	C	E	N	E
	Corogard	E	E	N	C

NOTE: Corrosion on panels is in dissimilar areas.

E - Exfoliation C = Pitting Corrosion N = No visible Corrosion

Exposure Period = 21 Months

CONCLUSIONS

5.3 Optimum Surface Treatment and Coatings

The coatings and corrosion evaluations in this program indicated the following:

- 5.3.1 The optimum surface treatment is phosphoric acid anodize. The primed panels of phosphoric and chromic acid anodize performed equally in all tests except in the exfoliation corrosion test. In this test the phosphoric anodize panels exhibited a much greater resistance to general and exfoliation corrosion than the chromic on all the alloys and tempers tested. It also reduced the degree of coating failures.
- 5.3.2 The optimum bonding primer is BR127. The comparison of the different bonding primers showed that all primers are compatible with any of the coating systems of Table 16; and, BR127 used under the coating systems reduced the degree of coating failure in dissimilar metal areas.
- 5.3.3 The optimum exterior coating system over the BR127 primer is MIL-P-23377 primer plus MIL-C-83286 topcoat (system C-2). This system appears to inhibit exfoliation better than Corogard (system C-5).
- 5.3.4 The optimum interior coating systems over BR127 are as follows:
 - ° Above the floor line is MIL-P-23377 primer (system C-1). Corrosion resistance is greatly improved.
 - ° Below the floor line is MIL-P-23377 primer plus MIL-C-83286 topcoat (system C-2). This is in preference over the MIL-P-23377 primer plus PR1432GP sealant primer plus the MIL-C-83019 clear overcoat (system C-3). The reasons are (1) any bond failure or corrosion initiating under system C-3 can not be easily detected visually, (2) ease of stripping may be a problem, (3) coating failure in dissimilar metal areas are more gross and (4) adhesion of the MIL-C-83019 clear to the PR1432GP is marginal to poor.

5.4 Testing of Selected Final Article Coating Systems

Protection of the bond joint provided by the interior and exterior coating systems that were selected was determined. Specimens were prepared and tested as noted in Table 31D. Lap shear specimens were scribed through the bond joint prior to environmental exposure to simulate a damage in the protective coating. Results are as follows:

5.4.1 Data for the lap shear strength data are shown in Tables 22, 23 and 24.

Shear strength values were not affected by any of the environments. Examination of the bond joint showed no corrosion initiating at the scribed line, and all lap shear failures were cohesive.

5.4.2 Corrosion Fatigue - The corrosion fatigue test was conducted on a control specimen (BR127 only) in a 3.5% saline solution at 1500 psi maximum and at $R = -0.2$. Ten million cycles were run with no failures occurring. Testing of the other topcoated specimens were discontinued since the control did not fail.

TABLE 22
STRENGTH OF LAP SHEAR SPECIMENS

ENVIRONMENT (60 DAYS)	COATING SYSTEM	SPEC. NO.	SHEAR STRENGTH (PSI)	TYPE FAILURE
Acidified Salt Spray	C-2	1	5490	Cohesive
		2	5557	
		3	5571	
		4	5616	
		5	5580	
	C-3	1	5545	Cohesive
		2	5684	
		3	5719	
		4	5916	
		5	5907	
Humidity	C-2	1	5748	Cohesive
		2	5444	
		3	5561	
		4	5873	
		5	5821	
	C-3	1	4663	Cohesive
		2	4801	
		3	4783	
		4	4773	
		5	4736	
Bilge Fluid	C-2	1	6261	Cohesive
		2	6076	
		3	6217	
		4	6242	
		5	6174	
	C-3	1	5623	Cohesive
		2	5846	
		3	5842	
		4	5892	
		5	6038	
Salt Spray	C-2	1	5885	Cohesive
		2	5837	
		3	5752	
		4	5924	
		5	5927	
	C-3	1	5333	Cohesive
		2	5436	
		3	5443	
		4	5320	
		5	5331	
None	BR127 (Control)	1	5839	Cohesive
		2	5828	
		3	5772	
		4	5514	
		5	5377	

Alum. Alloy - Nonclad 2024-T3
Anodize - Phosphoric Acid

Bond Primer - BR127
Adhesive - FM73

TABLE 23
STRENGTH OF LAP SHEAR SPECIMENS

ENVIRONMENT (60 DAYS)	COATING SYSTEM	SPEC. NO.	SHEAR STRENGTH (PSI)	TYPE FAILURE
Acidified Salt Spray	C-2	1	6460	Cohesive
		2	6322	
		3	6304	
		4	6011	
		5	6496	
	C-3	1	5612	Cohesive
		2	5677	
		3	5573	
		4	4288	
		5	5581	
Humidity	C-2	1	4588	Cohesive
		2	5927	
		3	5915	
		4	5853	
		5	5923	
	C-3	1	5682	Cohesive
		2	6119	
		3	6360	
		4	6226	
		5	6072	
Bilge Fluid	C-2	1	6613	Cohesive
		2	6437	
		3	6422	
		4	6441	
		5	6408	
	C-3	1	5915	Cohesive
		2	5990	
		3	5982	
		4	6376	
		5	6172	
Salt Spray	C-2	1	5525	Cohesive
		2	5839	
		3	5825	
		4	5700	
		5	5986	
	C-3	1	5754	Cohesive
		2	5978	
		3	5792	
		4	5873	
		5	6461	
None	BR127 (Control)	1	6008	Cohesive
		2	5691	
		3	5630	
		4	6000	
		5	6132	

Alum. Alloy - ~~Alclad 7075-T6~~
Anodize - Phosphoric Acid

Seal Primer - BR127
Inhibitor - 1017

TABLE 24
STRENGTH OF LAP SHEAR SPECIMENS (BEACH EXPOSURE)

ALUM. ALLOY (NONCLAD)	COATING SYSTEM	SPEC. NO.	SHEAR STRENGTH (psi)		TYPE FAILURE
			6 MO.	12 MO.	
2024-T3	C-2	1	5182	5938	Cohesive
		2	5244	5822	
		3	5564	5986	
		4	5608	5920	
		5	5489	6046	
	C-3	1	5387	5492	Cohesive
		2	5454	5594	
		3	5813	5376	
		4	5819	6039	
		5	5729	5834	
	BR127 (Control) Unexposed	1	5839	—	Cohesive
		2	5828		
		3	5772		
		4	5614		
		5	5977		
7075-T6	C-2	1	6293	5845	Cohesive
		2	6349	5445	
		3	6267	5707	
		4	6628	5791	
		5	6520	5417	
	C-3	1	6132	6719	Cohesive
		2	6042	5651	
		3	5874	5466	
		4	5711	5330	
		5	5923	5370	
	BR127 (Control) Unexposed	1	6008	—	Cohesive
		2	5091		
		3	5030		
		4	6000		
		5	6132		

Anodize - Phosphoric Acid, Bond Primer-BR127, Adhesive-FH73

Exposure Period = 6 & 12 months, El Segundo Beach Site.

5.5 Protection of Trimmed Edges

The effectiveness of edge sealing trimmed edges in protecting the bond line from corrosion was determined. Specimens were prepared and tested as noted in Table 31D. The edges of the BR127/FM73 bonded specimens were trimmed, treated with Alodine 1200S prior to edge sealing with the four sealant/urethane topcoat systems. Results are as follows:

- 5.5.1 Specimens were split open at the bond joint and inspected after 10 months in the bilge fluid and after 12 months on the beach. No corrosion was observed along the trimmed edges and all adhesive failures were cohesive. The only coating system degradation observed on the bilge fluid specimens was on the specimens sealed with the PR1436G sealant. They exhibited numerous blistering along the edges. Of the systems exposed on the beach, the MIL-C-83019 clear applied over the PR1432GP had flaked and lifted. It also exhibited cracking over the PR1436G. The MIL-C-83286 topcoat was also cracked over the PR1436G.

5.6 Clad Versus Nonclad Alloys

The effects of the environment on phosphoric acid anodized clad and nonclad aluminum assemblies were determined. Specimens were prepared and tested as noted in Table 31D. A panel from each set was fillet sealed at the edges and the bond joints with MIL-S-61733 sealant (PR1436G). Results are as follows:

- 5.6.1 Specimens, both sealed and unsealed, were pulled apart and the bond joint inspected.
- a. Acidified Salt Spray (12 months) - The maximum exposure time in this environment has normally been 2000 to 3000 hours. Exposure for the bonded butt face panels was extended to 12 months since they did not exhibit any apparent visible bond deterioration. This was an extremely long time in a severe environment. Of the panels tested, the specimens cut from the unsealed nonclad 7075-T6 panel, were the only ones having failures. Two out of five specimens failed. There was corrosion that had progressed inward from the unsealed edge into the bond joint. It is suspected that due to the warpage in the component panels that made up this particular butt face specimen, inadequate pressure was obtained at the bond joint causing a defective bond.

None of the other specimens had any corrosion failures and all adhesive failures were cohesive.

- b. Beach exposure (12 months) and bilge fluid exposure (6 months) specimens all passed without any failures.

5.7 Coating Rework

The effects of the two methods (organic strippers and grit blasting) of coating removal and/or subsequent rework on the mechanical bond line properties and on coating adhesion were determined. Specimens were prepared and tested as noted in Table 31D. Results are as follows:

5.7.1 The results indicate:

- a. Data for the strippability of BR127 are shown in Table 25. None of the phenolic or acid strippers were effective in removing properly applied and cured BR127 from a phosphoric acid anodized surface.
- b. Data for the strippability of the protective coating systems applied over BR127 are shown in Table 26. Turco 5351 brush on stripper was used.
- c. Data for the shear strength values after exposure to a stripper and to grit blasting are shown in Table 27. Shear strength values were not affected by any of the removal methods.
- d. Butt face specimens with stripper residue left in the bond line and overcoated with the exterior coating system were exposed on the beach for 6 months. Specimens were pulled apart and inspected. There was no corrosion in the joint and all adhesive failures were cohesive.
- e. Adhesion of the exterior coating system (C-2) to a reworked (grit blast and alodine 1200S) phosphoric acid anodize surface exhibited cohesive failure in the MIL-P-23377 primer.

5.8 Finish Document

The "PABST Finish Specification" and F-527 "PABST Full Scale Development Component" finish specifications establishing the minimum requirements necessary to provide adequate protection from corrosion for the PABST components and fuselage were written. See Appendix.

TABLE 25
STRIPPING OF BR127 PRIMER

STRIPPER	TYPE	EXPOSURE TIME	EFFECT ON BR127
Turco 5351	Phenolic	8 hrs.	None - Sl. Discoloration
Turco 6017	Acid	8 hrs.	None - Sl. Discoloration
See Lee A-202	Acid	8 hrs.	None - Sl. Discoloration
E. Resin ES-	Acid	8 hrs.	None - Sl. Discoloration
Chem. Acid (hot)	Acid	1/4 hr.	Complete removal

TABLE 26
STRIPPING OF PROTECTIVE COATINGS

COATING SYSTEM OVER BR127	STRIPPABILITY (1)		
	COATING	TIME (MIN)	REACTION
MIL-P-23377 Primer	MIL-P-23377	35	Slight Softening
		90	Partial Removal
MIL-P-23377 Primer + MIL-C-83285 Topcoat	MIL-C-83285	10	Complete Removal
MIL-P-23377 Primer + PR1432 GP Sealant + MIL-C-83019 Topcoat	MIL-P-23377	40	Slight Softening
	MIL-P-23377	90	Partial Removal
		90	Partial Removal
MIL-P-23377 Primer + PR1432 GP Sealant + MIL-C-83019 Topcoat	MIL-C-83019	2	Complete Removal
PR1432 GP Sealant + MIL-C-83019	PR1432 GP	30	Slight softening and partial removal down to MIL-P-23377 primer.
	MIL-P-23377	90	Partial Removal
		90	Partial Removal
PR1432 GP Sealant + MIL-C-83019	MIL-C-83019	2	Complete Removal
	PR1432 GP	30	Slight softening and partial removal.
		90	Removed down to BR127.
Corogard	Corogard	15	Complete removal down to BR127.

FOOTNOTE: (1) All systems required scraping with a wooden scraper and repeated application of stripper (Turco 5351).

All systems using MIL-P-23377 primer over the BR127 were removed down to the MIL-P-23377 primer. The MIL-P-23377 primer softened only slightly. All systems without the MIL-P-23377 primer were stripable down to the BR127 primer. The PR 1432 GP sealant softens slightly and requires repeated scraping and stripper application for removal.

TABLE 27
SHEAR STRENGTH OF LAP SHEAR SPECIMENS

Method	COATING REMOVAL		SPEC. NO.	SHEAR-STRENGTH (PSI)	TYPE FAILURE
	MEDIA	EXPOSURE TIME			
Stripping	Turco 5351	2 hrs.	1	4932	Cohesive
			2	4131	
			3	4372	
			4	4732	
			5	4308	
Grit Blasting	Aluminum Oxide (150 grit)	3 minutes ①	1	4540	Cohesive
			2	4842	
			3	4766	
			4	4735	
			5	4804	
	Walnut Shell (20-30 grit)	10 minutes ①	1	4360	Cohesive
			2	5006	
			3	5286	
			4	4985	
None	None (control)	None	1	4618	Cohesive
			2	4969	
			3	4725	
			4	4452	
			5	4538	

NOTE: Primer - BR127, Adhesive - FM73

① Grit blasting time is dependent on removal of the coating system down to the metal substrate.

5.9 BR127 Versus BR127A Adhesive Primers

A second version of BR127 was known to exist and was in use by one of the large aircraft companies. The second version was identified as BR127A and was said to have better low temperature peel properties than BR127. A comparison of the BR127 and BR127A primers was made to determine if any differences exist in their film properties and in corrosion resistance. Specimens were prepared and tested as noted in Table 31E. Results are as follows:

5.9.1 Data for the fineness of grind, adhesion, salt spray, immersion corrosion and exfoliation corrosion tests are shown in Table 28.

- a. The fineness of grind of the BR127A produced a smaller particle size than the BR127.
- b. Dissimilar metal panels after 4 weeks in the acidified salt spray showed the following:
 - ° Coating failures around dissimilar metal areas were slightly more for the BR127A compared to BR127 batch 564 and batch 613.
 - ° In the primer only test, corrosion pits appeared earlier on the BR127A (batch 1180) and BR127 (batch 613) coated phosphoric acid anodize surfaces than on the BR127 (batch 564). Batch 1180 exhibited more corrosion pits than batch 613.
 - ° No exfoliation was present on any of the panels at 4 weeks.
 - ° In addition, a new batch of BR127A batch 1181 was also tested. BR127 batch 613 was used as the control. These two primers have the same solvent composition blend in the formulation. The only difference being a variance in the ratio of the resins used. Corrosion tests results of batch 1181 were similar to that previously obtained with the BR127A batch 1180. BR127A provides less corrosion protection to the phosphoric acid anodize surface than BR127.
- c. Results of the other tests showed BR127A and BR127 to be comparable.

TABLE 28
COMPARISON OF BR127 PRIMERS

PROD.	BOND PRIMER			OVERCOAT SYSTEM	SALT SPRAY (2000 HRS)	ADHESION (WET)	IMMERSION CORR.			ACIDIFIED SALT SPRAY (4 WKS)
	BATCH	GRID	THICK. (MILS)				CORR.	ADH.	OTHER FAIL	
BR127	564	7	.12	None	P	P	②	P	None	No Exfoliation
				C-2	P	P	None	P	None	
				C-3	P	F①	None	F①	Blist #8 Scott	
BR127A ③	1180	4	.18	None	P	P	②	P	None	No Exfoliation
				C-2	P	P	None	P	None	
				C-3	P	F①	None	F①	Blist #8 Scott	④
BR127	613	7	.15	None	NOT TESTED					No Exfoliation

NOTE: P = Pass, F = Fail

- ① Adhesion failure of the clear topcoat to PR1432GP sealant.
- ② Corrosion in scribe line with filament type corrosion extending out from scribe.
- ③ The applied film of BR127A is gritty and appears to be either resin or chromate agglomerates.
- ④ Coating failures around dissimilar metal areas were slightly more for the BR127A compared to BR127.

5.10 MEK Resistance of BR127 Adhesive Primer

During the production phase of the PABST program, it became apparent that the cured primer on some details was easily removed by wiping with a cloth dampened in methyl ethyl ketone than other primed and cured details. This condition posed a problem, not in the bond joint where subsequent curing in the presence of the adhesive film would complete the cure of the primer, but in the areas elsewhere on the assembly where the primer would be covered with a paint film and become part of the corrosion protection systems for the base alloys.

This evaluation was conducted to determine the causes of failure, when wiped with an MEK dampened cloth, in BR127 and the subsequent corrosion resistance of finish systems which have uncured BR127 as the initial primer coat.

Specimens were prepared and tested as noted in Table 31C.

- a. The matrix for attempting to reproduce the MEK wipe failures of BR127 was completed using three different batches of the primer. The batches were #636 (3/8/76), #651 (5/3/76) and #654 (5/12/76). The primers were siphon spray applied in the same manner as accomplished in production. The following spray equipment, the atomizing pressures and the fluid needle adjustments were used for preparing the panels.

Equipment - DeVilbiss JGA gun, FX fluid tip and needle and #36 air can.
Atomizing pressure at the gun - 40 and 60 psi.
Fluid needle adjustment - full open and half open.
- b. Panels for the environmental tests of uncured BR127 were processed as follows: The three batches of BR127 were applied and processed to provide the cured and uncured conditions as determined in the Table 29 matrix. A set of cured and uncured panels were each coated with and without the MIL-P-23377 primer and overcoated with the MIL-C-83286 topcoat. Prior to overcoating all cured BR127 panels were cleaned with MEK. All uncured panels were cleaned with 1,1,1 trichloroethane. Another set of uncured panels were MEK cleaned and the BR127 completely removed prior to applying the MIL-P-23377 plus MIL-C-83286 system.

Chemical analyses were also conducted on three different batches of BR127 to determine whether differences existed. American Cyanamid's quality control procedures were used in these analyses.

Results are as follows:

MEK Resistance Matrix of BR127 - The primer batch numbers, curing temperatures, cure times, the required and actual film thicknesses, the atomizing pressure/fluid needle combination, and the MEK resistance are all noted in Table 29.

Data indicate that high or normal atomizing pressures and low or normal fluid needle adjustments are not causes for poor MEK resistance. It appears that the problem may be attributed to differences in primer batches in conjunction with a low film thickness application of 0.1 mil or less. As noted in Table 29, the curing of primer at 200°F either failed or exhibited solvent attack as expected when subjected to an MEK wipe. However, batch 636 was the only batch that exhibited removal by MEK after 225° and 250°F cure cycles.

TABLE 29
MEK RESISTANCE OF BR127

CURE TEMP. (°F)	REQ. THICK (MILS)	CURE TIME (HRS.)	BATCH 636			BATCH 651			BATCH 654		
			A	B	C	A	B	C	A	B	C
250	.1	3/4	.1 F	.15 F	.12 F	.12 P	.08 P	.1 P	.1 P	.1 P	.1 P
		1	.1 F	.08 F	.1 F	.1 P	.12 P	.12 P	.1 P	.15 P	.08 P
	.3	3/4	.2 P	.28 P	.35 P	.28 P	.3 P	.3 P	.3 P	.35 P	.28 P
		1	.2 P	.25 P	.35 P	.25 P	.3 P	.3 P	.32 P	.38 P	.25 P
225	.1	3/4	.15 P	.18 P	.1 F	.15 P	.15 P	.18 P	.1 P	.1 P	.1 P
		1	.13 P	.15 P	.12 P	.15 P	.13 P	.1 P	.12 P	.12 P	.1 P
	.3	3/4	.4 P	.38 P	.35 P	.3 P	.4 P	.38 P	.3 P	.3 P	.25 P
		1	.38 P	.38 P	.35 P	.32 P	.4 P	.35 P	.3 P	.32 P	.25 P
200	.1	3/4	.12 F	.08 F	.05 F	.1 F	.1 F	.1 F	.12 F	.15 F	.08 F
		1	.1 F	.12 PS	.05 F	.1 PS	.1 PS	.08 PS	.1 PS	.1 PS	.08 PS
	.3	3/4	.25 PS	.3 PS	.28 F	.32 PS	.32 P	.25 PS	.32 PS	.32 PS	.35 PS
		1	.25 PS	.28 PS	.25 PS	.35 PS	.32 P	.25 PS	.35 PS	.32 PS	.28 PS

P = Pass F_m = Fail PS = Pass but MEK dulls primer film

► Atomization pressure/fluid needle adjustment

A = 80 psi/full open

B = 80 psi/half open

C = 40 psi/full open

c. Environmental tests data showed the following:

Wet adhesion tests were conducted on all sets without any adhesion failures.

Exfoliation corrosion testing in the acidified salt spray environment was terminated after 3000 hours. None of the dissimilar metal panels exhibited any evidence of exfoliation corrosion. All had general corrosion and coating failure of the three batches of BR127. The amount of corrosion on the uncured BR127 (all three batches) specimens overcoated with only the MIL-C-83286 topcoat is greater than any of the other specimens. The amount of failure of the other specimens within each batch of BR127 were comparable to each other except as noted above.

Table 29 shows the difference in corrosion inhibition, even though slight, between the three batches. The decreasing order of inhibition is from batch 654, 651 to 636.

None of the dissimilar metal panels exposed on the beach showed any coating failures or corrosion after 12 months except for the uncured BR127 (batch 636) panel overcoated with only the MIL-C-83286 topcoat.

d. Chemical analysis results are shown in Table 30.

TABLE 30
CHEMICAL ANALYSIS

ANALYSIS	CONTROL VALUES	BR127 BATCH NO.		
		636	651	654
Oxirane Ratio	0.78 \pm .08%	1	0.86	0.71
Total Solids	10 \pm 1%	10.1	10.5	10.7
Percent Pigment	-	13.4	17.1	15.5
Solvent Comp.	(% by vol.)			
MEK	65	63	62	57
THF	20	17.5	19.5	24.7
DAA	15	13.5	11.9	13.8
Others	-	6	6.6	4.5
IR of cured versus uncured films	-	No significant diff.		

NOTE: Curing agent determination was not conducted due to problems in completely digesting the primer sample.

The oxirane ratio (see Figure 72 - Transmission at 910 cm^{-1} and 810 cm^{-1}) of batch 636 is much higher than batch 651 or 654. Whether this has a significant effect on the curing is not known.

The IR analysis of the cured versus uncured films indicated no detectable differences in the curves of any of the batches. However, an IR curve of a cured and an uncured film of batch 636 taken from a production run indicated differences in absorption in the 1620 cm^{-1} region.

The percent pigment in batch 636 is the lowest of the three batches. This difference can be visually seen on the panels sprayed at 0.1 mils and upon observing the pigment layer in the container after it had settled.

The solvent composition of batch 654 differs the most from the control values. There is a significant change in the amount of MEK and THF. It was noted that batch 654 stays in suspension much longer than either batch 636 or 651.

BR127 batch 636 exhibited repeated MEK resistance failures on parts processed in production. MEK failures were usually on parts having film thicknesses of about 0.1 mil. Other batches have also been applied in production at similar thicknesses without exhibiting any MEK failures.

A sample of batch 636 was given to American Cyanamid for analysis. It was reported that this batch was definitely low in the amount of curing agent. The exact amount was not given.

In summary, it has been shown that BR127 films of 0.1 mil or less is very susceptible to removal by wiping with MEK. Not having the necessary amount of curing agent contributed appreciably to this problem.

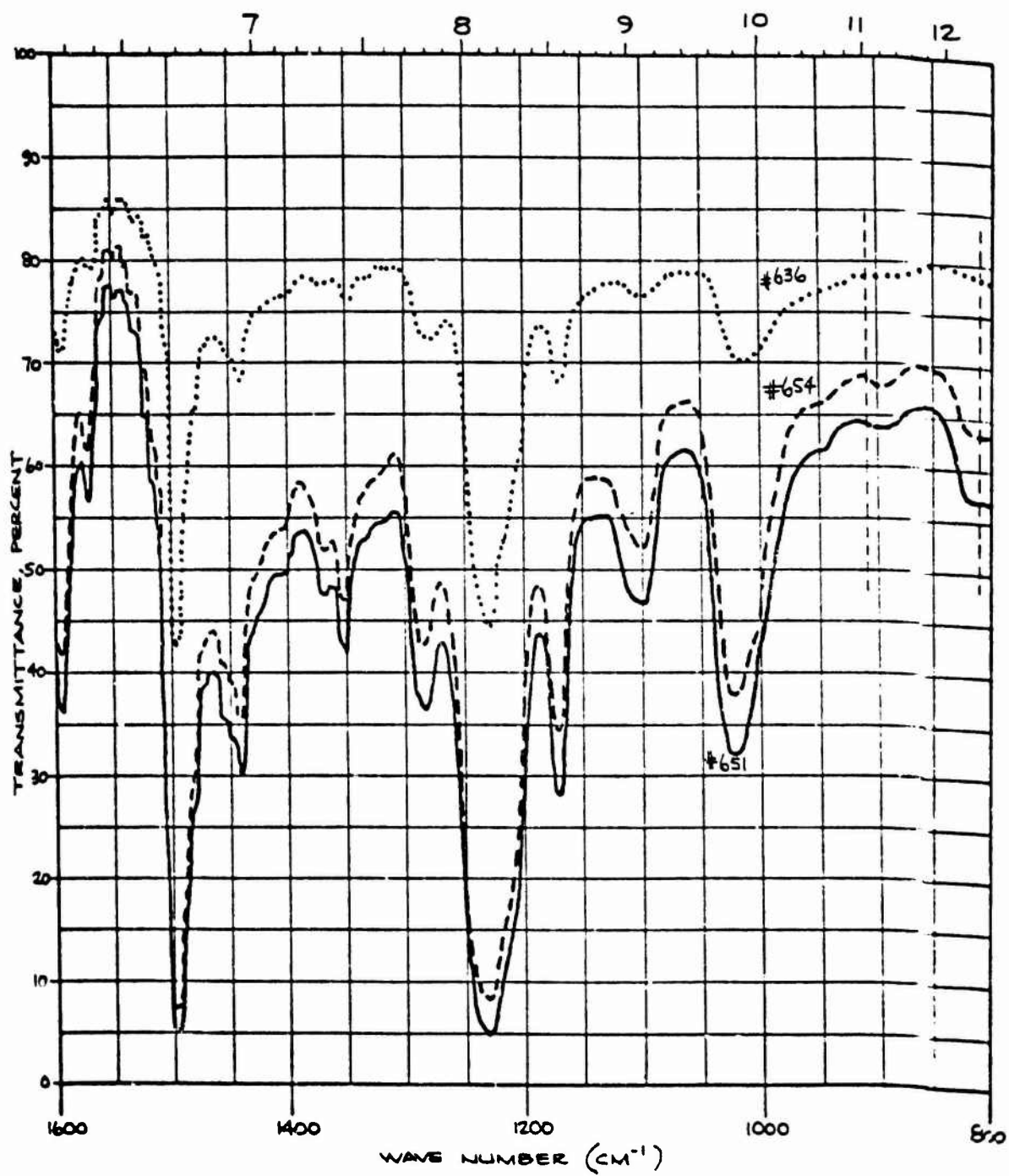


FIGURE 72. OXIRANE RATIO OF BR127

BATCH	CURED BR 127		UNCURED BR 127		UNCURED BR127 + MEK REMOVED
	MIL-P-23377 + MIL-C-83286	MIL-C-83286 ONLY	MIL-P-23377 + MIL-C-83286	MIL-C-83286 ONLY	MIL-P-23377 + MIL-C-83286
636					
651					
654					

Exposure Period = 3000 Hours

FIGURE 73. ACIDIFIED SALT SPRAY - CURED VERSUS UNCURED BR127

TABLE 31A
SPECIMEN PREPARATION AND TESTS

IDENTIFI- CATION	TEST SPECIMEN		ALUMINUM ALLOY		SURFACE TREATMENT (TABLE 13)	COATING	SALT SPRAY (TABLE 32)
	TYPE	FIG.	NONCLAD	GAUGE			
Corr. Resist. of Surface Treatments	Flat	74-11	7075-T6	.125	Phosphoric Acid Anodize	None	X
						BR127	X
					MDC Chromic Acid Anodize	None	X
						BR127	X
					Bell Helicopter Chromic Acid Anodize	None	X
						BR127	X
					FPL Etch	None	X
						BR127	X
Corr. Resist. of the MDC Chromic Acid Anodize Sealing Matrix	Flat	74-11	2024-T3	.125	MDC Chromic Acid Anodize	None	X
						BR127	X

TABLE 31E
SPECIMEN PREPARATION AND TESTS

TEST SPECIMEN		ALUMINUM ALLOY		SURFACE TREATMENT (TABLE 13)	COATING SYSTEM (TABLE 1)	ENVIRONMENTAL TESTS (TABLE 32)						IMMER. CORR.	BILGE FLUID	
TYPE	FIG.	HINICLAD	GUAGE			SALT SPRAY	EXFOLIATION	BLACH	HUMIDITY	ADHESION				
Adhesive/ Bond Primer	74-4	7075-T6	.032	Phosphoric Acid Anodize	C-1	X				X	X	X		
					C-2	X				X	X	X		
					C-3	X				X	X	X		
					C-4	X				X	X	X		
					C-5	X				X	X	X		
Flat	74-1		.032		C-1						X	X		
					C-2							X	X	
					C-3							X	X	
					C-4							X	X	
					C-5							X	X	
Fastener (Bonded)	74-2		.125		C-1			X						
					C-2			X						
					C-3			X						
					C-4			X						
					C-5			X						
Fastener/ Doubler	74-7				C-2									
					C-5						X	X		

TABLE 31C
SPECIMEN PREPARATION AND TESTS

TEST SPECIMEN		ALUMINUM ALLOY		SURFACE TREATMENT (TABLE 13)	COATING SYSTEM (TABLE 1)	ENVIRONMENTAL TESTS (TABLE 32)						IMMER. CORR.	BILGE FLUID
TYPE	FIG.	NONCLAD	GAUGL			SALT SPRAY	EXFOLIATION	BEACH	HUMIDITY	ADHESION			
Flat	74-1	2024-T3 7075-T6	.032	Phosphoric Acid Anodize & Chromic Acid Anodize	C-1	X			X	X		X	X
					C-2	X				X			
					C-3	X			X	X		X	X
					C-4	X			X	X		X	X
					C-5	X					X		
Fastener (Bonded)	74-2		.125		C-1		X						
					C-2		X						
					C-3		X						
					C-4		X						
					C-5		X						
Fastener/ Doubler	74-7				C-2			X					
					C-5		X						
					C-1						X		
					C-3						X		
					C-4						X		
Flat	74-1	7075-T73 & T76	.125		C-1							X	
					C-3							X	
					C-4							X	
					C-2								
					C-5								
Fastener/ Doubler	74-7		.125		C-2			X					
					C-5		X						
					C-1								
					C-2								
					C-5								
Fastener (Bonded)	74-2		.125		C-1		X						
					C-2		X						
					C-3		X						
					C-4		X						
					C-5		X						

TABLE 31D
SPECIMEN PREPARATION AND TESTS

TEST SPECIMEN		ALUMINUM ALLOY		SURFACE TREATMENT (TABLE 13)	COATING SYSTEM (TABLE 1)	ENVIRONMENTAL TESTS (TABLE 32)					EILGE FLUID	STRIPPING
TYPE	FIG.	UNSCLAD	GAUGE			SALT SPRAY	EXFOLIATION	BEACH	HUMIDITY	ADHESION	CORR. FATIGUE	
Lap Shear	74-5	2024-T3 7075-T6	.063	Phosphoric Acid Anodize	C-2 C-3	X X			X X			X X
Corr. Fatigue	74-3		.125		C-2 C-3	X X		X X				
Trimmed Edge	74-3	7075-T6	.063	Phosphoric Acid Anodize	C-4 C-6 C-7 C-8			X X X X			X X	
Butt Face	74-6	7075-T6 7075-T3 & Clad 7075-T6	.125	Phosphoric Acid Anodize	C-2	X	X	X			X	
Lap Shear	74-5	2024-T3 7075-T6	.125	Phosphoric Acid Anodize	C-3	X	X				X	
Butt Face	74-6		.125		C-2 C-3							X X
Flat	74-1		.063		C-2 C-3			X X				

TABLE 31E
SPECIMEN PREPARATION AND TESTS

TEST SPECIMEN		ALUMINUM ALLOY		SURFACE TREATMENT (TABLE 13)	COATING SYSTEM (TABLE 1)	ENVIRONMENTAL TESTS (TABLE 32)				
TYPE	FIG.	NONCLAD	GAUGE			HEK RESIST.	ADHESION	INER. CORR.	EXFOLIATION	SALT SPRAY
Flat	74-1	7075-T6	.032	Phosphoric Acid Anodize	C-2		X	X		X
					C-3		X	X		X
Fastener (Bonded)	74-2		.125		C-2				X	
					C-3				X	
Flat	74-1	7075-T6	.032	FPL Etch Phosphoric Acid Anodize	BR127	X				
					C-2		X			
Fastener	74-9		.125		C-2				X	
Fastener	74-10		.125		C-2					
										X

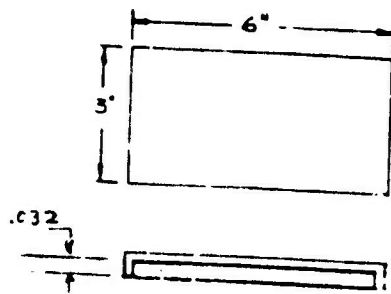


FIGURE 74-1. FLAT

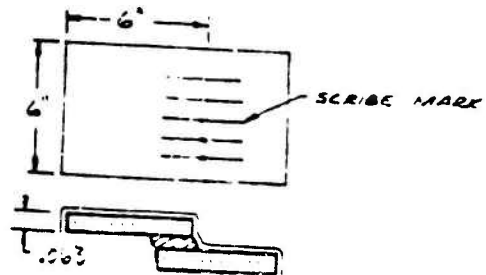


FIGURE 74-5. LAP SHEAR

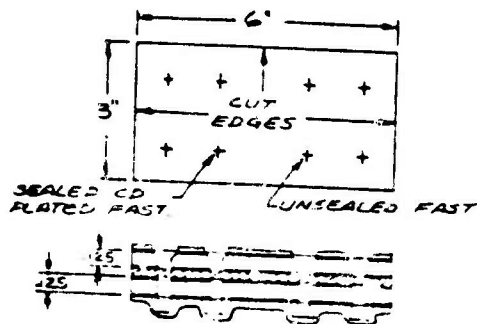


FIGURE 74-2. FASTENER (BONDED)

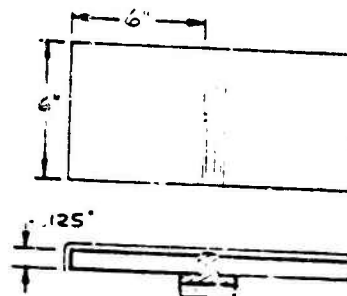


FIGURE 74-6. BUTT FACE (SEALED AND UNSEALED AT BONDLINE WITH PR1436G)

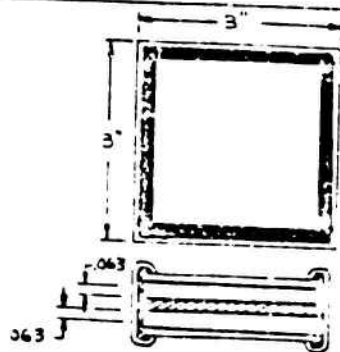


FIGURE 74-3. TRIMMED EDGE

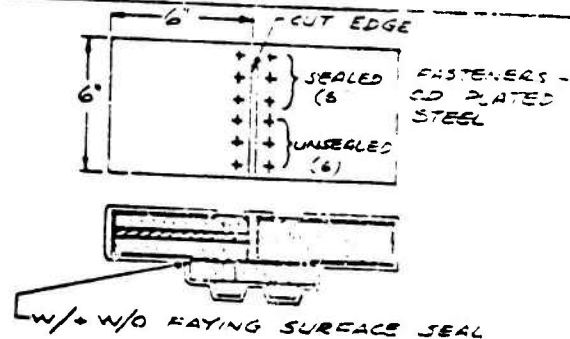


FIGURE 74-7. FASTENER/DOUBLER

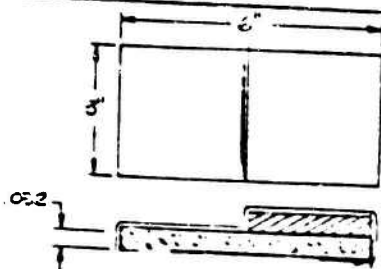


FIGURE 74-4. ADHESIVE/BOND PRIMER

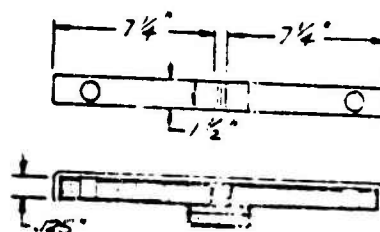




FIGURE 74-8. CORROSION FATIGUE

 Metal Substrate
 Adhesive

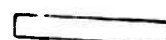

 Coating
 Sealant

FIGURE 74A. TEST SPECIMEN CONFIGURATIONS

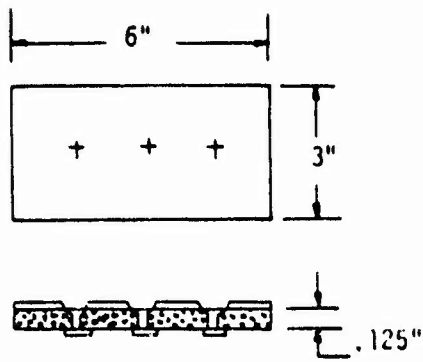


FIGURE 74-9. FASTENER

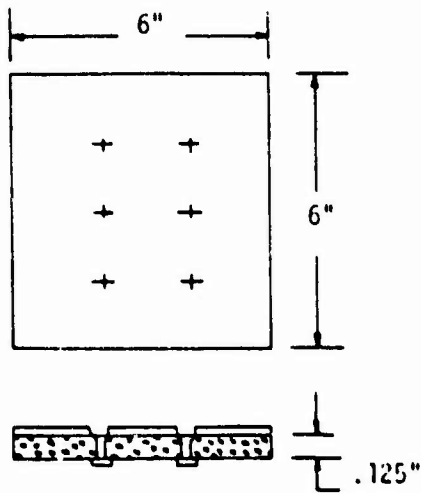


FIGURE 74-10. FASTENER

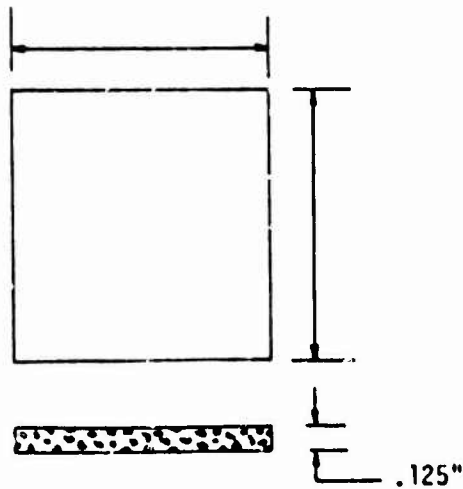


FIGURE 74-11. FLAT

FIGURE 74B. TEST SPECIMEN CONFIGURATIONS

TABLE 32
SUMMARY OF TESTS

TEST	TEST CONDITIONS*
Salt Spray Corrosion	FTMS 141, Method 6061. 5-Percent NaCl. Expose 2000 Hours.
Exfoliation Corrosion	5-Percent NaCl Solution Acidified to pH of 3 using Acetic Acid. Tower Temperature 112 to 125°F, Cabin Temperature 100 to 118°F. Cycle = 45-Minute Spray, 120-Minute Purge, 195-Minute Dwell. Expose as required.
Beach Site Exposure	El Segundo Beach Rack. Expose as Required.
Immersion Corrosion	5-Percent NaCl Acidified to pH of 3 using Acetic Acid. Immersion at RT. Expose 30 Days.
Corrosion Fatigue	1500 psi Load, R=0.2 Load Rate, Immersion at RT in 3.5-Percent NaCl Solution. Expose Million Cycles.
Humidity	95 ± 5% RH, 120 ± 2°F. Expose 30 Days.
Adhesion	FTMS 141, Method 6201 Except Scribe "X" Between the two Parallel Scribes. Expose 24 Hours at RT.
Gilge Fluid	Sodium Sulfate/Sodium Chloride in Distilled Water. pH of 7.8. 30 Days Immersion at RT.

* FTMS refers to Federal Test Methods Standards

- NOTES:
1. Salt Spray Corrosion - Panels were diagonally scribed through the coating down to the basis metal. They were exposed to the salt spray at an angle of 5° from the vertical for 2000 hours. Examination made at 500 hour intervals or as deemed necessary. Evidence of general corrosion and blistering, corrosion extending out from the scribe mark, loss of adhesion and other film failures were noted.
 2. Exfoliation Corrosion - Panels were exposed to the acidified salt fog at an angle of 45°. Examinations were made at weekly intervals to determine the ability of the various coating systems to inhibit exfoliation and general corrosion.
 3. Beach Site Exposure - Panels were exposed on the beach rack at El Segundo, California. Examinations were made at monthly intervals. Evidence of general corrosion, lifting, exfoliation, filiform, coating degradation and blistering were noted.

4. Immersion Corrosion - Panels were scribed through the coating down to the basis metal. They were immersed in 5% sodium chloride acidified to pH 3 for 30 days. Evidence of corrosion and film failures were noted.
5. Corrosion Fatigue - Panels were fatigue tested in a saline environment for a million cycles. Time to failure was determined.
6. Humidity Resistance - Panels were hung vertically in a 90-100% condensing humidity atmosphere for 30 days. Panels were examined at weekly intervals and noted for blistering, lifting and other film failures. Adhesion was determined at the end of the test period.
7. Adhesion - The dry adhesion was determined using 3M #250 pressure sensitive tape. Wet adhesion was determined by immersion in distilled water for 24 hours. Immediately after removal from the water the wet adhesion was determined. Panels were examined for blistering and other film failures.
8. Bilge Fluid - Panels were scribed through the coating down to the basis metal. They were immersed at RT in the fluid for 30 days. Evidence of corrosion and film failures was noted.

SECTION VI ADHESIVES

6. Introduction

Part of the adhesive selection criteria has been mentioned in the introduction to this compilation. To iterate, the selection of the candidate adhesives to be tested was based on the operating parameters that would be expected to be experienced in the final article to be fabricated. These parameters include an elevated temperature exposure of 180°F, wet humid conditions, and -70°F exposure. Several adhesive systems are available that can produce good bonds and are durable under these conditions.

6.1 Material Selection

In this program, it was decided to test a series of new modified epoxy film adhesive systems that cure at 250°F (121°C) and have a corrosion resistant adhesive primer system. The adhesives selected for test are listed in Section 5.1 and represent some of the best available adhesives in this category.

The tests in this section (6) were mechanical tests and were performed on the adhesives to determine if any of the basic properties of any of the systems could be considered unacceptable and reject the system before environmental tests and long term tests could be started. They also formed a base for properties that could be used in preliminary design studies of the aircraft structure.

The selected surface treatment used in this part of the program is the phosphoric acid anodize. The standard processing procedures were used on the anodize to reduce the number of variables that had to be considered in the adhesive testing part of the program.

Each adhesive system was ordered from the vendors as their standard product to meet the requirements of "state-of-the-art." Each vendor was requested to supply a corrosion adhesive primer that they considered adequate for the test program. A requirement for the type of carrier used with the adhesive was not made and, as a consequence, three of the adhesives were supplied with a woven type carrier and one was supplied with a Dacron mat type carrier. In the tests performed in this program, it can be seen the effect of the type of carrier. Any effort for other Engineering designs should consider all the aspects of the materials properties and tests designed or selected that will best represent the problem to be resolved.

6.2 Materials and Processes

This section lists the materials and processes used to fabricate the test specimens. The phosphoric acid anodize was used for all of the test specimens. The processing procedure was standard and is covered in Section 2 of this report.

The processing procedures for the adhesive primers and the adhesives were obtained from the vendors data sheets. All times, temperatures and pressures were maintained to meet these standard procedures. Processing parameter investigations were conducted in another part of the program.

6.2.1 Adhesive Systems

Four major suppliers of aircraft structural adhesives were requested to submit samples of their latest state-of-the-art new generation 250°F epoxy adhesive systems. These systems were intended to be new low-moisture permeable, corrosion inhibiting adhesives. No specific carrier for the adhesive was requested.

The following adhesive systems and batch numbers were used for material evaluation:

MATERIAL	TYPE	BATCH NO.	MANUFACTURER
FM73 NONWOVEN CARRIER	ADHESIVE FILM 0.045 LB/SQ FT	BATCH 86 ROLL 326	AMERICAN CYANAMID
BR127	PRIMER	BATCH 560	AMERICAN CYANAMID
EA9628 WOVEN CARRIER	ADHESIVE FILM 0.045 LB/SQ FT	BATCH 076-134 ROLL D	HYSOL DIVISION OF THE DEXTER CORP
EA9202	PRIMER	BATCH 103-186	HYSOL DIVISION OF THE DEXTER CORP
AF55 WOVEN CARRIER	ADHESIVE FILM 0.045 LB/SQ FT	BATCH 1D5P	MINNESOTA MINING AND MANUFACTURING (3M)
XA3950	PRIMER	BATCH 3B5P	MINNESOTA MINING AND MANUFACTURING (3M)
M1133 WOVEN CARRIER	ADHESIVE FILM 0.045 LB/SQ FT	BATCH 30 ROLL 1	NARMCO MATERIALS, INC
M6740	PRIMER	BATCH 17	NARMCO MATERIALS, INC

6.2.2 Primer Application and Cure

The primers were removed from 0°F storage. They were allowed to come to ambient temperature before opening and were thoroughly shaken to ensure all solids were in suspension. They were applied within 2 hours after the phosphoric acid anodize oven dry and were continuously agitated during application. A wet uniform cross coat was applied to each faying surface using a DeVilbiss JGA spray gun with FX fluid tip and needle and a #36 air cap, with air pressure of 45 ± 5 psi at the spray gun and fluid needle full open. The primer was applied to a dried film thickness of 0.0001 to 0.0002 inch as measured with an Isometer film thickness measurement device. The primed details were air dried 30 minutes followed by an oven dry of 60 minutes at 225°F.

6.2.3 Adhesive Application and Cure

The adhesive films were removed from 0°F storage and allowed to come to ambient temperature before the opening of the protective wrappings. One layer of adhesive film was used in each bond line. All metal to metal specimens were shimmed during layup to dry and maintain a uniform .005 inch bond line. All specimens were assembled and cured within 8 hours after phosphoric acid anodizing (laboratory control requirement). Specimens were vacuum bagged on a 0.250 inch aluminum plated with a 0.250 inch aluminum caul plate used on top of specimens to try to obtain uniform bond lines. An exception to this was the 0.500 inch thick double cantilever beam specimens. Specimens were cured in the laboratory autoclave. The chamber pressure is raised to 40 psi and then the temperature was increased at a rate of 3-5 degrees per minute to the maximum temperature of $245 \pm 5^{\circ}\text{F}$. The parts are held at pressure and maximum temperature for 90 minutes. Temperatures are monitored at the bond line.

6.3 Mechanical Properties

Mechanical property tests were run using several types of specimens. These tests were intended to give general overall properties of the adhesive systems prior to the beginning of the environment resistance tests. The processing of the adherends, priming and curing of the specimens were as described in Section 5.11. The number of specimens required, general material gauge and test conditions are specified in Tables 33 and 34.

TABLE 33
MECHANICAL PROPERTIES -- ADHESIVES

SPECIMEN TYPE	SPECIMEN DRAWING	ALLOY	ALLOY THICKNESS	LAP LENGTH	ADHESIVE	NO. OF SPECIMENS			REMARKS
						-70	RT	180	
LAP SHEAR	SEE FIGURE 1	7075-T6 BARE	0.063	0.50	FM73	5	5	5	TOTAL SPECIMENS 10
					EA9628	5	5	5	
					AF55	5	5	5	
					M1133	5	5	5	
DOUBLE LAP SHEAR	SEE FIGURE 2	7075-T6 BARE	0.063	0.50	FM73	5	5	5	TOTAL SPECIMENS 10
					EA9628	5	5	5	
					AF55	5	5	5	
					M1133	5	5	5	
T-PEEL	SEE FIGURE 3	2024-T3 BARE	0.032	TEST AREA 1 IN. x 9 IN.	FM73	5	5	5	TOTAL SPECIMENS 10
					EA9628	5	5	5	
					AF55	5	5	5	
					M1133	5	5	5	
CREEP	SEE FIGURE 4	7075-T6 BARE	0.063	LAP LENGTH 0.50	FM73		5	5	TOTAL SPECIMENS 40
					EA9628		5	5	
					AF55		5	5	
					M1133		5	5	
CLIMBING DRUM	SEE FIGURE 5	7075-T6 BARE	0.020	TEST AREA 3 IN. x 9 IN.	FM73	5	5	5	TOTAL SPECIMENS 60
					EA9628	5	5	5	
					AF55	5	5	5	
					M1133	5	5	5	

TABLE 34
MECHANICAL PROPERTIES - ADHESIVES

SPECIMEN TYPE	SPECIMEN DRAWING	ALLOY	ALLOY THICKNESS	TEST AREA	LAP ADHESIVE	NO. OF SPECIMENS			REMARKS
						-70	RT	180	
FLATWISE TENSION	SEE FIGURE 6	7075.T6 BARE	0.020	2 IN x 2 IN.	FM73		5	5	TOTAL SPECIMENS 40
					EA9628		5	5	
					AF55		5	5	
					M1133		5	5	
BEAM SHEAR	SEE FIGURE 7	7075.T6 BARE	0.020	8 IN x 4 IN.	FM73		5		TOTAL SPECIMENS 20
					EA9628		5		
					AF55		5		
					M1133		5		
THICK ADHEREND	SEE FIGURE 8	7075.T6 BARE	0.50	LAP LENGTH 0.50	FM73	5	5	5	TEST NOT TO FAILURE FOR DETERMINATION OF ADHESIVE MODULUS. TOTAL SPECIMENS 60
					EA9628	5	5	5	
					AF55	5	5	5	
					M1133	5	5	5	
NAPKIN RING TEST	SEE FIGURE 10	7075.T6 BARE		TEST AREA APPROX 1.50 IN.	FM73		6		18 SHEAR MODULUS 18 TENSION MODULUS
					EA9628		6		
					AF55		5		
DOUBLE CANTI LEVER	SEE FIGURE 9	7075.T6 BARE	0.50	1 IN x 14 IN	FM73		5		TO BE COMPARED WITH 100% RH EXPOSED SPECIMENS TOTAL SPECIMENS 20
					EA9628		5		
					AF55		5		
					M1133		5		

6.3.1 Test Descriptions

6.3.1.1 Tensile Lap Shear

This test is a basic test to compare adhesive shear strength with the other adhesives in the program and adhesive systems generally used for this type of construction. It also indicated that the four adhesive systems could maintain an adequate shear stress over the temperature extremes that the final article would be exposed to.

The specimen configuration is shown in Figure 75. The test results are in Figure 82.

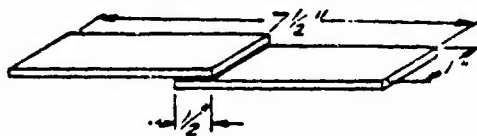


FIGURE 75. LAP SHEAR SPECIMEN

6.3.1.2 Double Lap Shear

The double lap shear specimens is similar to the lap shear except the eccentricity generated in the single lap shear specimen as it is stressed is reduced or eliminated. Since the specimens had .500 inch over lap, the bond area was twice that of the single overlap specimens and the specimens tested at room temperature and -70°F failed the metal. At 180°F , the average shear stress was slightly lower than for the single over lap shear specimens. Paradoxically, the lower peel adhesion system was least affected by the eccentricity produced by the single over lap shear test. Specimen configuration for the double lap shear specimens is shown in Figure 76. The test results are shown in Figure 83.

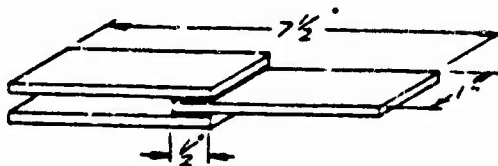


FIGURE 76. DOUBLE LAP SHEAR SPECIMEN

6.3.1.3 T-Peel

Metal to metal T-peel specimens were fabricated for each of the adhesive systems. Several methods are available for peel specimens in industry. The T-peel has been in use at Douglas for many years and is generally used in production as an overall production quality control specimen. T-peel strength generally indicates adhesive toughness and resistance to edge tension and flexing of the bonded joint. The test data indicates that the type of carrier has a large effect on the peel strength. The adhesive systems used in this program were specifically picked to have adequate peel strength at the minus temperatures where some adhesive systems become brittle, such as the nitrile-phenolics, and are subject to fracturing when flexed or fatigued. Of the three adhesives tested which had woven carriers, all had a high peel strength at ambient and at 180°F. Two of the three woven carrier systems had large reductions in peel strength at the -70°F test temperature. The adhesive with the mat carrier had a relatively low peel strength at all the temperatures tested. The specimen configuration is shown in Figure 77. Test results are shown in Figure 84.

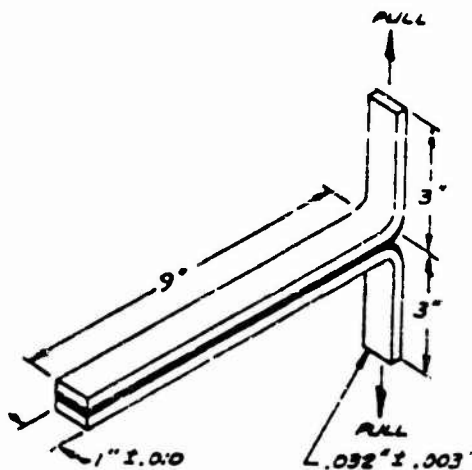


FIGURE 77. "T" PEEL SPECIMEN

6.3.1.4 Metal to Metal Creep

The creep test determines the amount of strain a lap shear specimen will move under a constant stress and constant temperature. The test was conducted on a standard over lap shear specimen. Test temperature was 180°F, load was 250 psi and the period of the test was 200 hours. No creep in the specimens was measurable. This information is confirmed by vendor data. The specimen configuration is shown in Figure 78.

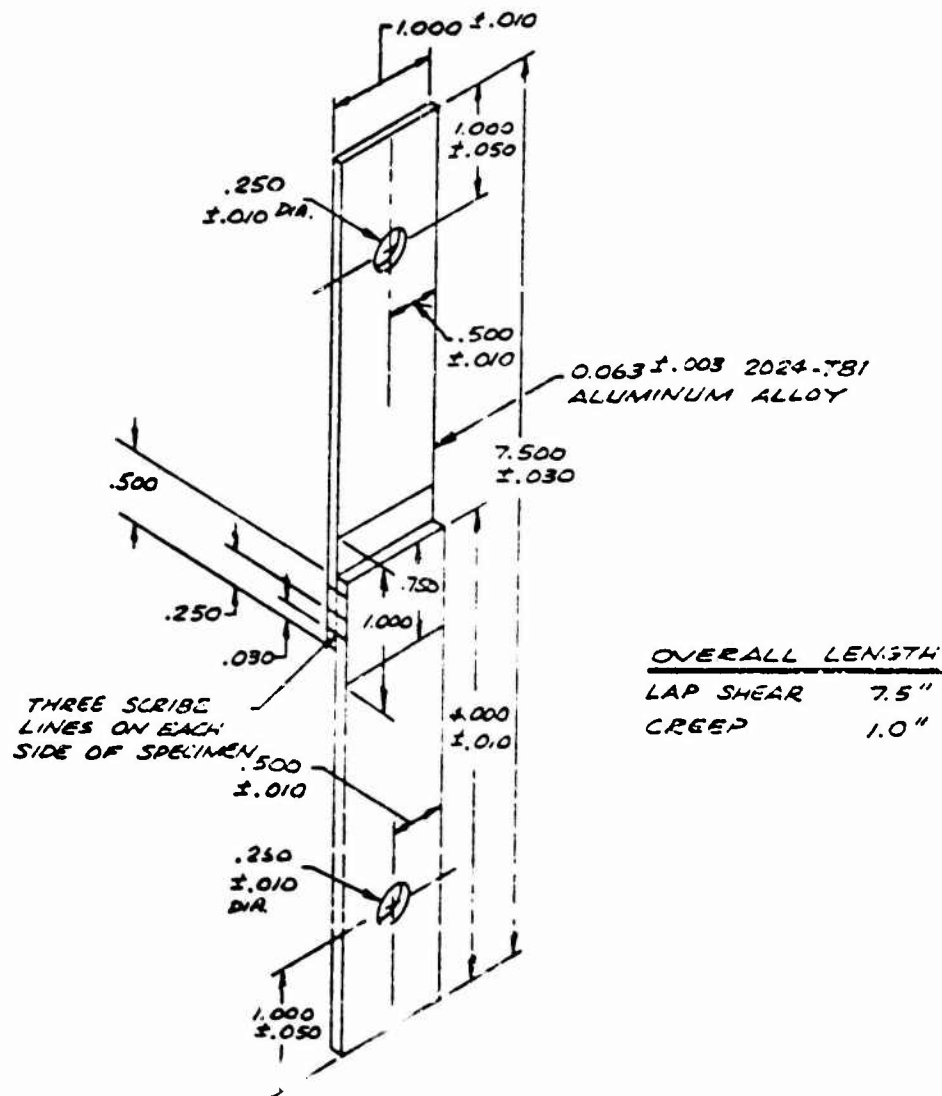


FIGURE 78. CREEP TEST SPECIMEN

6.3.1.5 Climbing Drum Peel

The original program had the possibility of having honeycomb structure in the design. Some testing was performed to meet this requirement. One of the standard test methods in industry is the climbing drum peel. This type of specimen gives information similar to the T-peel; i.e., a measure of toughness. It also predicts other properties that are important to honeycomb structure. This is to flow and fillet on the honeycomb cell wall in a manner to maximize the strength of the joint with a minimum of available adhesive. In the test, the adhesive system with the mat carrier generally had the lowest peel strength, as in the T-peel test. The three adhesives with woven carriers did not all respond similarly. One of the systems performed well below the others and at 180°F was lower than the mat carried system. It is apparent that before design can be considered, further testing of the systems is necessary to better characterize the adhesive. Specimen configuration is shown in Figure 79. Test results are in Figure 85.

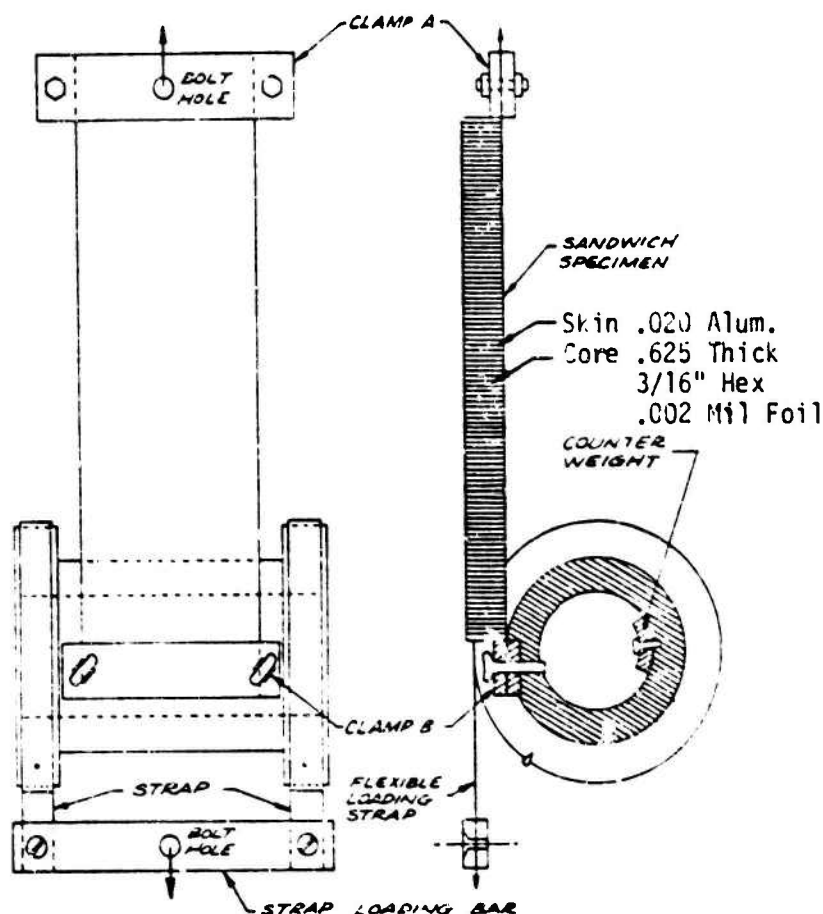


FIGURE 79. SANDWICH PEEL SPECIMEN AND TEST APPARATUS

6.3.1.6 Flatwise Tension

Flatwise tension tests also investigate adhesive properties on honeycomb. It determines the resistance to the tension failure and buckling of a skin being stabilized in a place by the honeycomb core. Adhesives with low peel strength and high tension strength have performed satisfactorily in structure, the results of this test indicate that all the adhesives in this program perform equally with the mat carried adhesives being the lowest in overall tension strength at the two temperatures tested. Specimen configuration is shown in Figure 80. The results are in Figure 86.

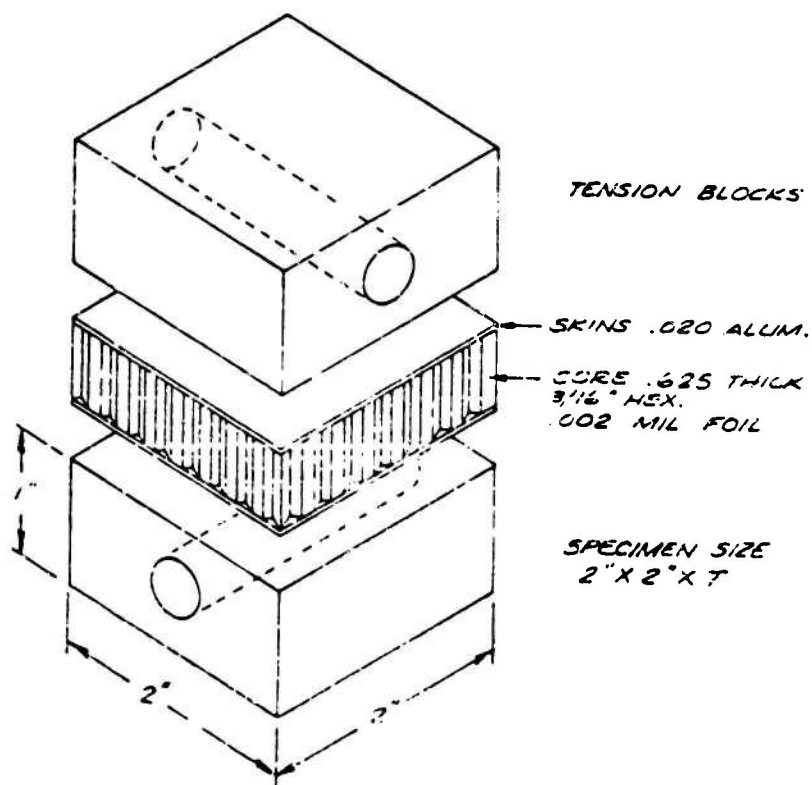


FIGURE 80. SANDWICH FLATWISE TENSILE SPECIMEN

6.3.1.7 Beam Shear

Beam shear tests, like the flat-wise tension tests, are a measure of the resistance of buckling of a skin bonded to honeycomb. It also can determine the shear strength of the adhesive at the skin/honeycomb interface and the shear strength of the honeycomb. These specimens were designed to put the highest stress on the skin/honeycomb interface. The results indicate the four adhesive systems performed equally. Specimen configuration is shown in Figure 81. Results are shown in Table 35.

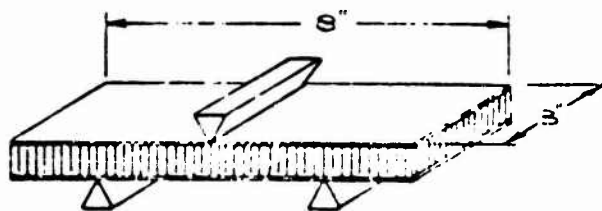


FIGURE 81. SANDWICH BEAM SHEAR SPECIMEN

TABLE 35
SANDWICH BEAM SHEAR

ADHESIVE SYSTEM	SPECIMEN IDENT.	SHEAR STRENGTH (psi)	FAILURE MODE
FM/ BR127	207-1-1	440	Core Shear
	-2	643	
	-3	641	
	-4	556	
	-5	561	
		<u>574</u> Avg.	
EA9628/ EA9202	207-2-1	521	
	-2	601	
	-3	611	
	-4	618	
	-5	593	
		<u>589</u> Avg.	
AF55/ XA3950	207-3-1	524	
	-2	506	
	-3	509	
	-4	495	
	-5	510	
		<u>509</u> Avg.	
M1133/ M6740	207-4-1	521	
	-2	561	
	-3	566	
	-4	570	
	-5	526	
		<u>549</u> Avg.	

Test Temp.: Ambient
 Materials: 7075-T6 Nonclad Alum. .020 inch Facings
 DMS 1588 Type 2 Grade B
 Core 6 lbs/ft³
 Adhesive Wt.: .045 lbs/ft²
 Surface Prep.: Phosphoric Acid Anodize

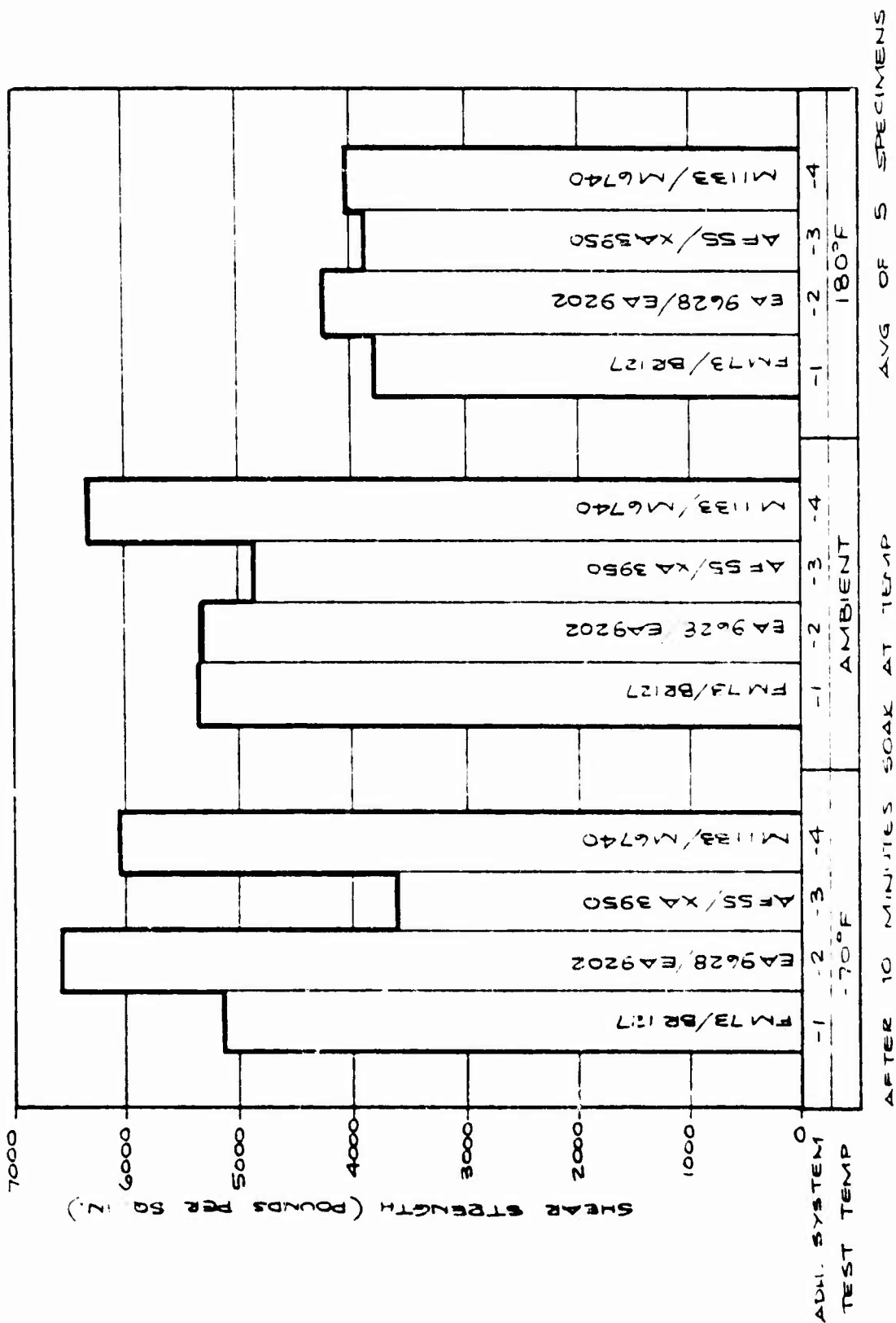


FIGURE 82. LAP SHEAR

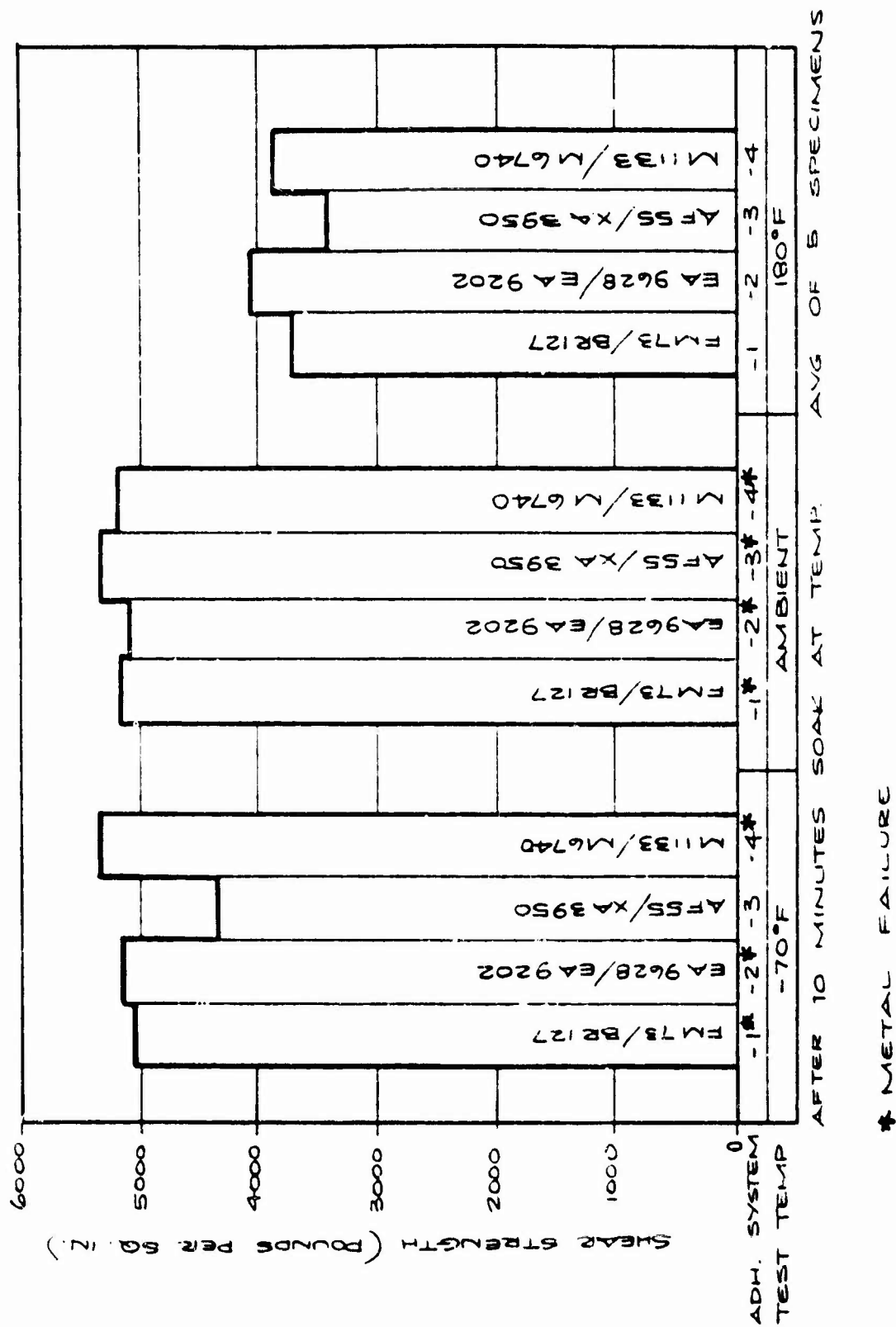


FIGURE 83. DOUBLE LAP SHEAR

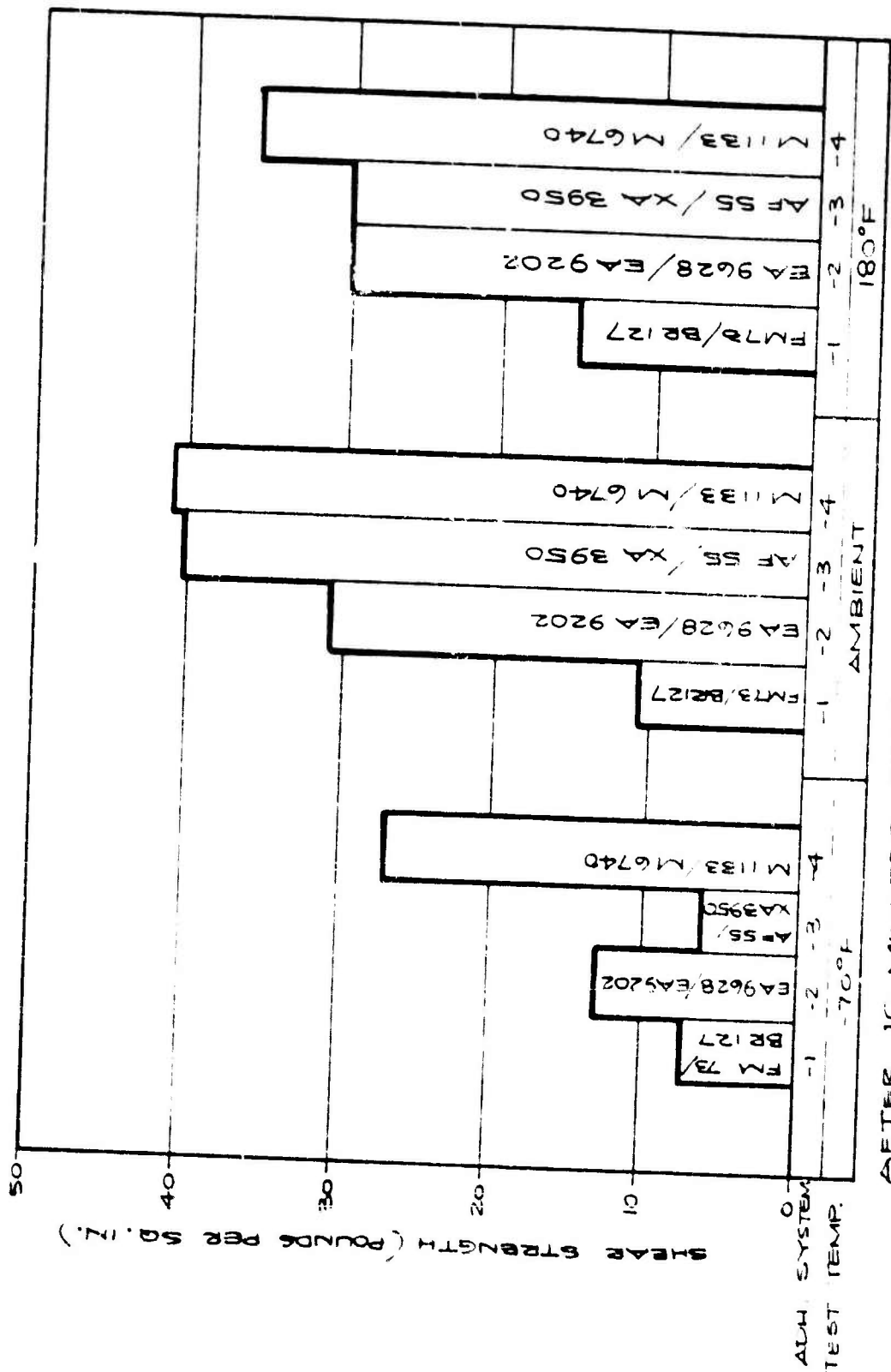
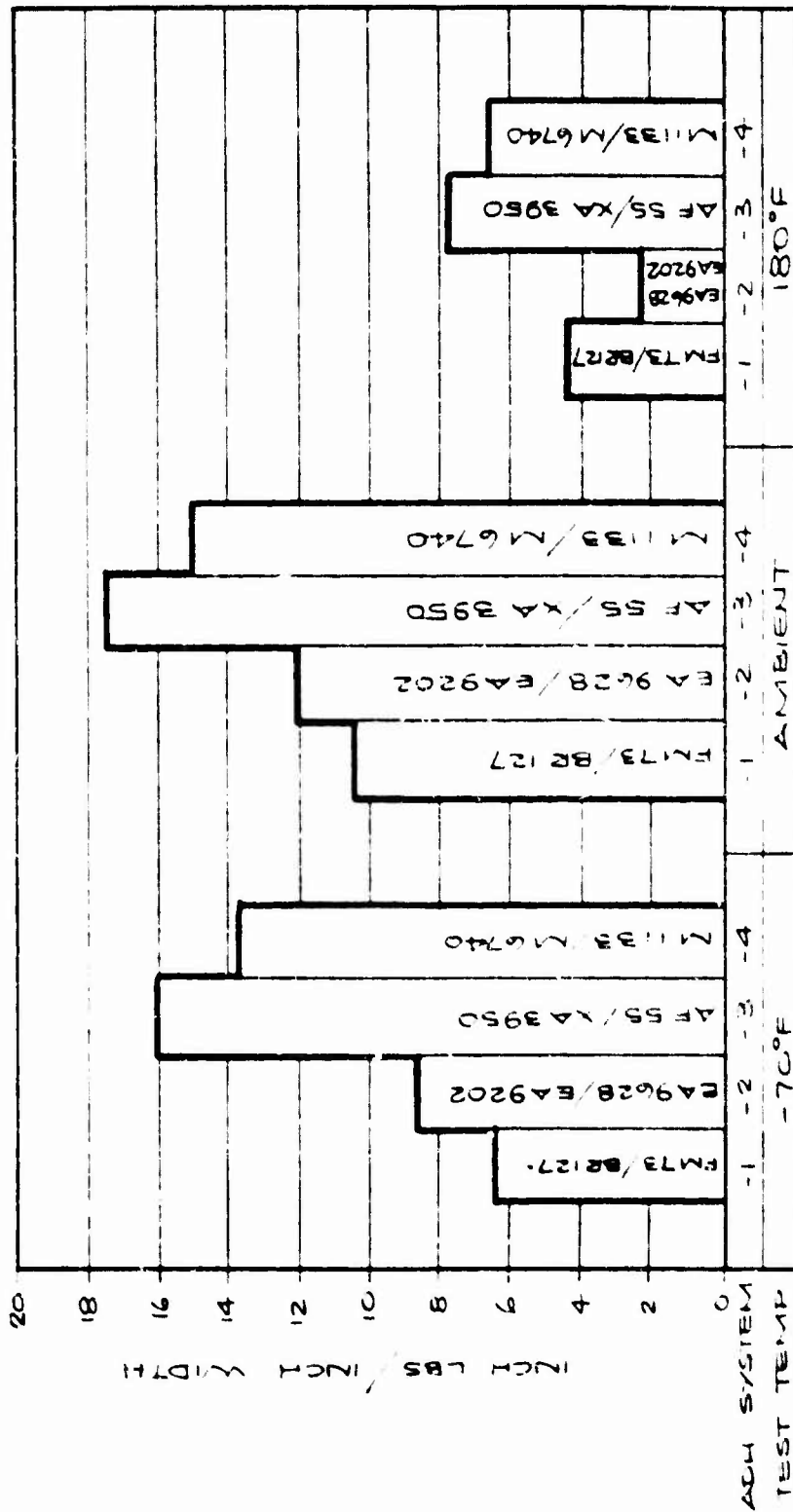
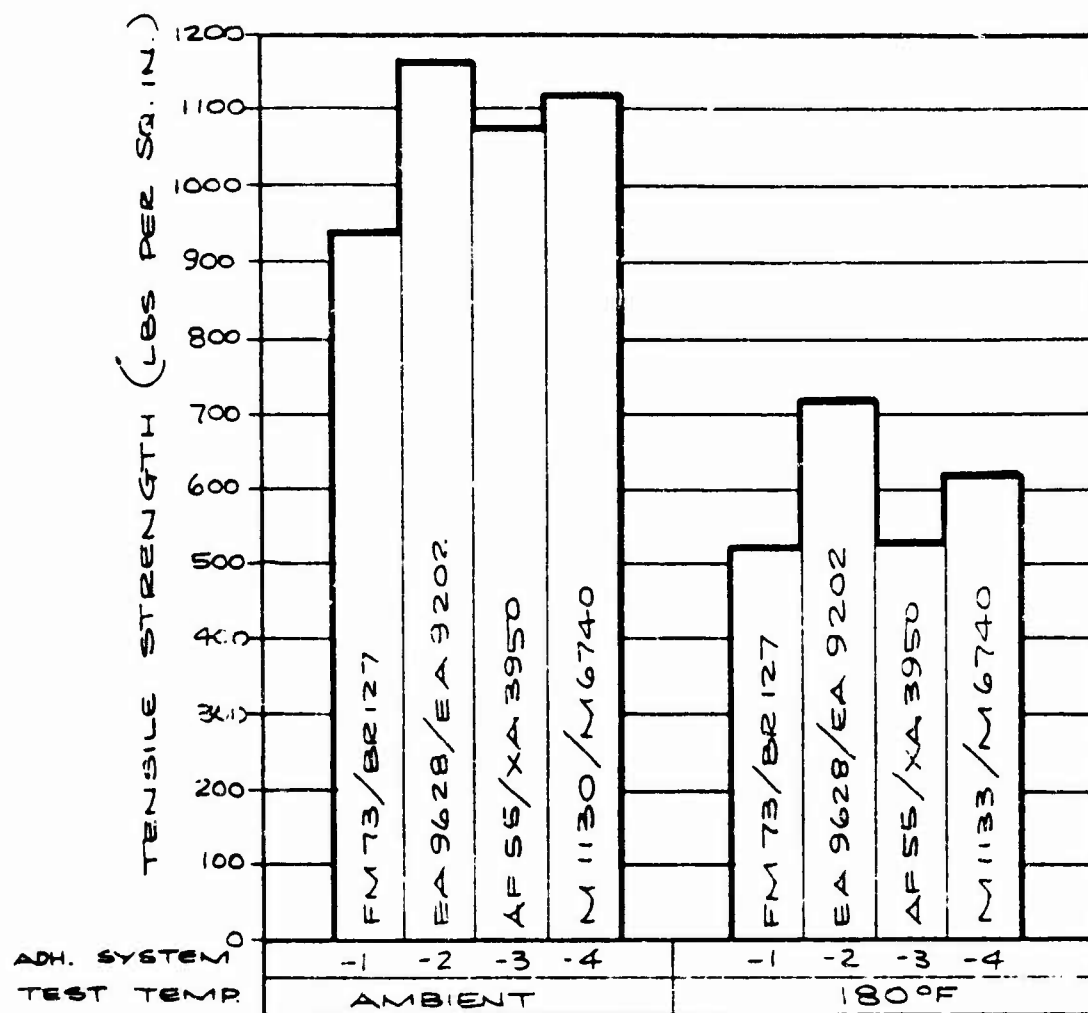


FIGURE 84. METAL TO METAL T-PEEL



AFTER 10 MINUTES SOAK AT TEMP AVG OF 5 SPECIMENS

FIGURE 85. SANDWICH CLIMBING DRUM PEEL



AFTER 10 MINUTE SOAK AT TEMP.
AVG OF 5 SPECIMENS

FIGURE 86. SANDWICH FLATWISE TENSION

6.3.1.8 Shear Modulus

Four methods were used to investigate adhesive deformation characteristics and shear modulus calculated for the linear range. These methods were:

- a. Thick adherend lap shear
- b. Napkin ring
- c. Torsion pendulum
- d. Neat adhesive tension

Figures 87, 88, 89, 90 and 91 illustrate the various test specimens and some of the type of test equipment for the four methods. Shear modulus was determined by inducing stresses in the thick adherend lap shear specimen and measuring the deformation using the zero gauge extensometer (Figure 88). Some difficulty was experienced with this test method since the measured deformation is very small. Extreme care is required to obtain consistent results. The instrument used in these tests was the KGR-1 supplied by American Cyanamid Co.

The napkin ring tests were performed by The Singer Company, Kearfott Division. The shear specimen consists of two thin walled tubes bonded end to end. They are stressed by applying a torsion load on the specimen. An extensometer measures the movement of one tube in relation to the others. Adherend deformation is also measured. The results in shear modulus determinations parallels that produced by the zero gauge extensometer on the thick adherend specimen.

The torsion pendulum tests were performed at The Netherland Aircraft Factories - Fokker-VFW, Amsterdam. The test apparatus is depicted in Figure 90. The specimen is cut from a sheet of precured adhesive one millimeter thick.

Neat adhesive tension tests were performed at strain rates of 0.00469 inch per inch per minute and 0.0469 inch per inch per minute, and load rates of 62 psi per minute to 62,000 psi per minute. Specimens were cut from precured adhesive, as shown in

Figure 91. Tests were also performed on preconditioned specimens representing fully wet bond lines.

Results of the tests are compared in Figures 92, 93, 94, 95 and 96.

The torsion pendulum and the neat adhesive tension tests showed similar moduli. All four adhesives tested showed similar mechanical properties and changes with temperature.

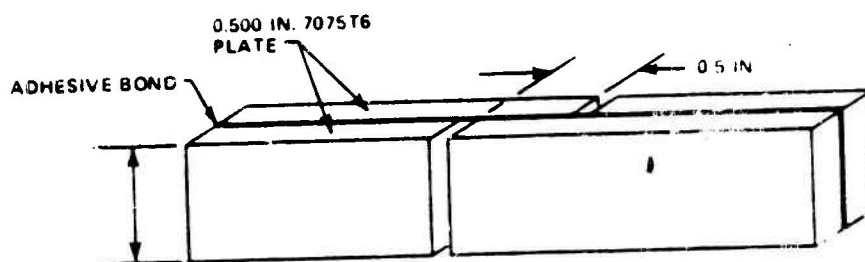
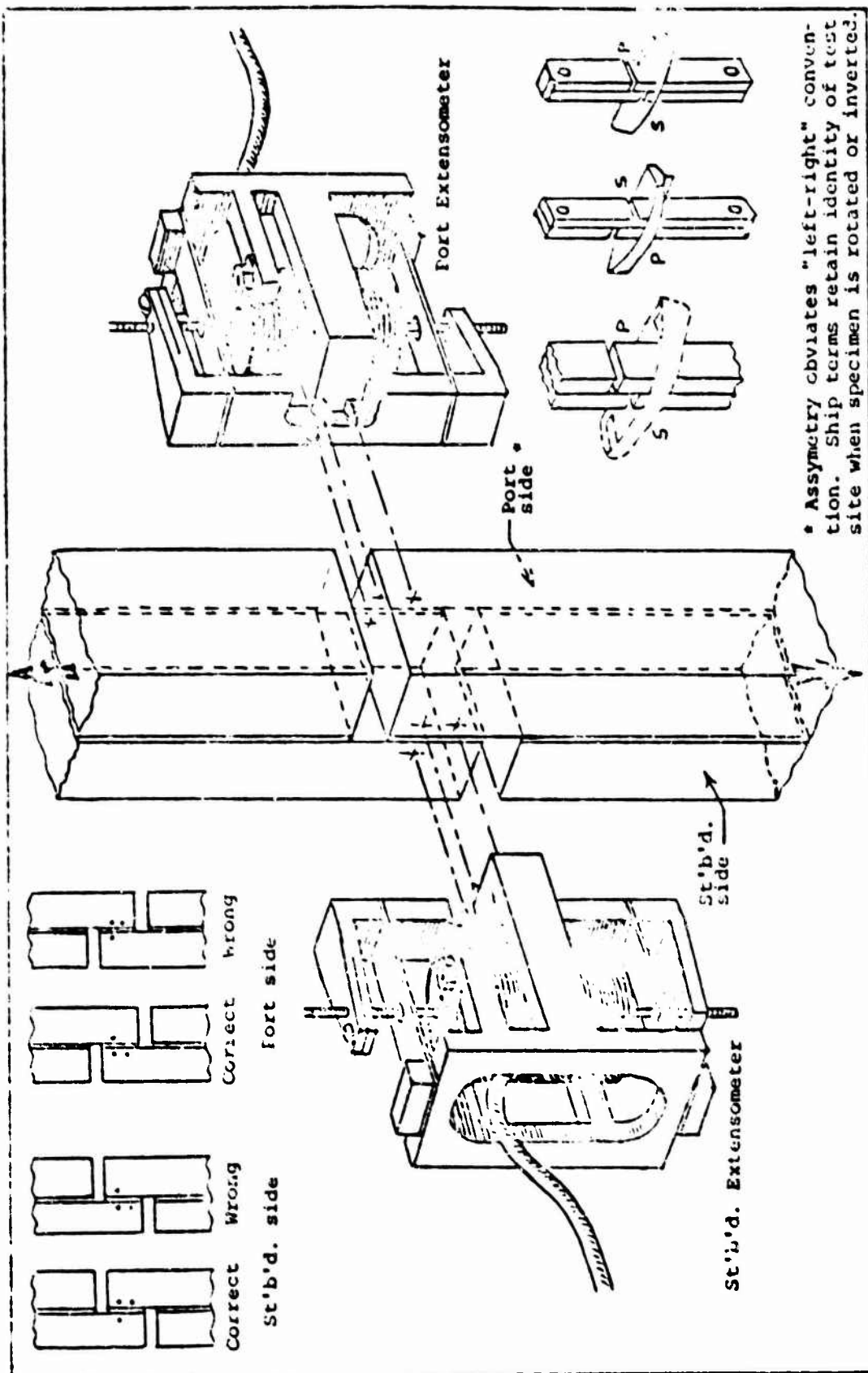


FIGURE 87. THICK ADHEREND TEST SPECIMEN



Courtesy American Cyanamid

FIGURE 88 SCHEMATIC OF THICK ADHEREND LAP SHEAR TEST APPARATUS

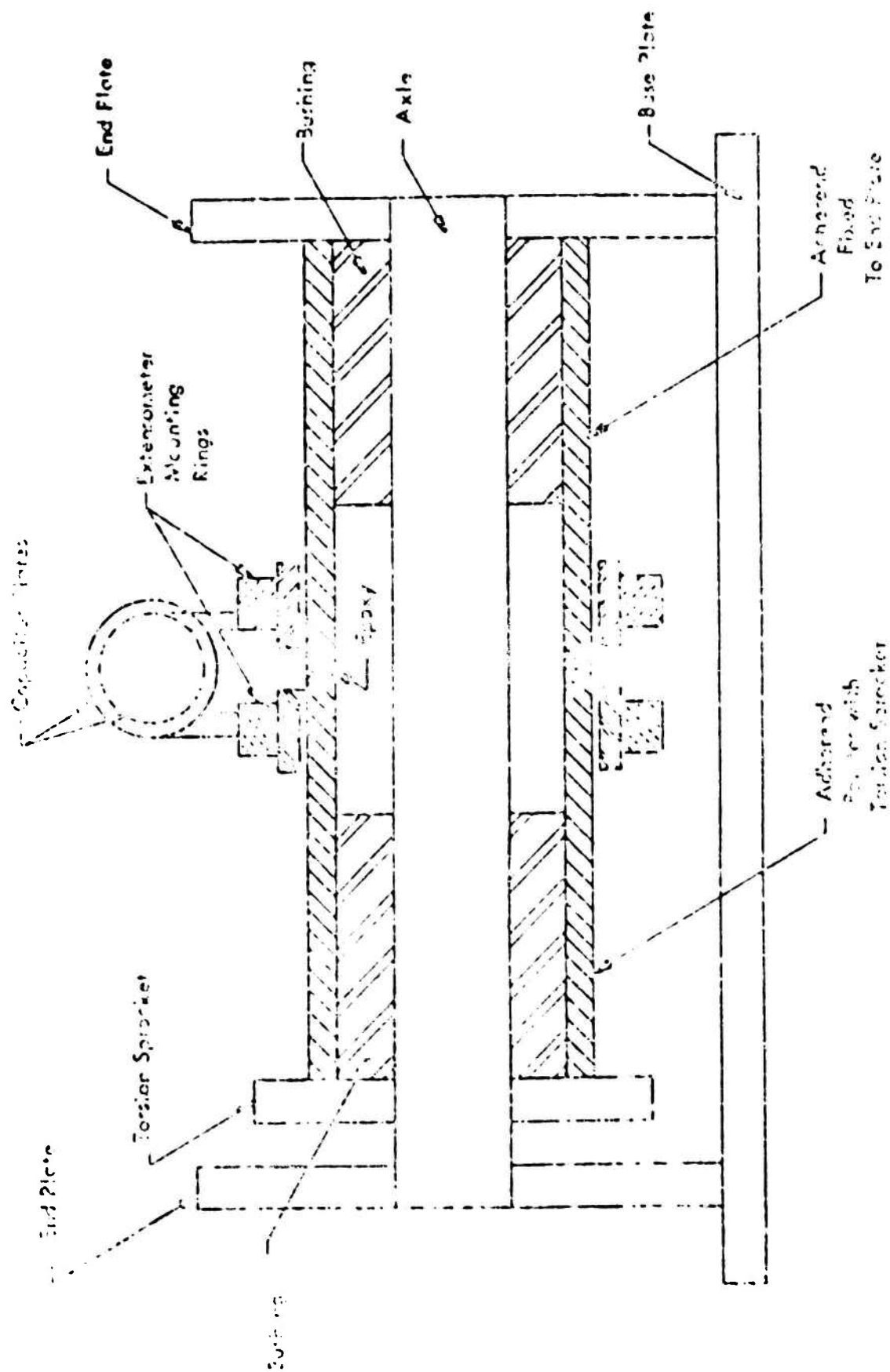
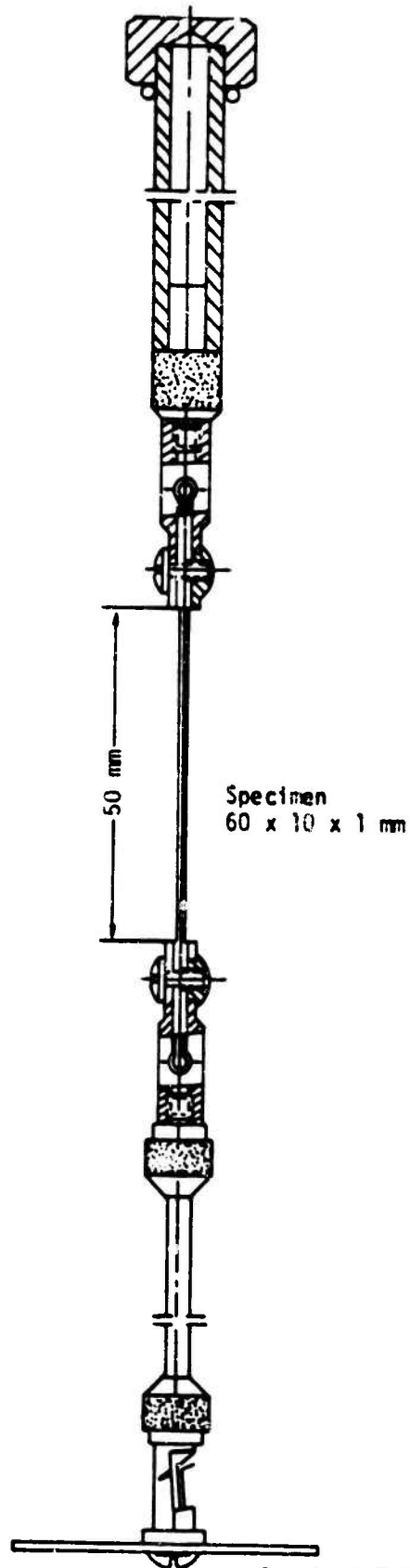


FIGURE 89. SCHEMATIC OF NAPKIN RING SHEAR TEST APPARATUS



Courtesy Fokker-VFW

FIGURE 90. SCHEMATIC OF TORSIONAL PENDULUM TEST APPARATUS

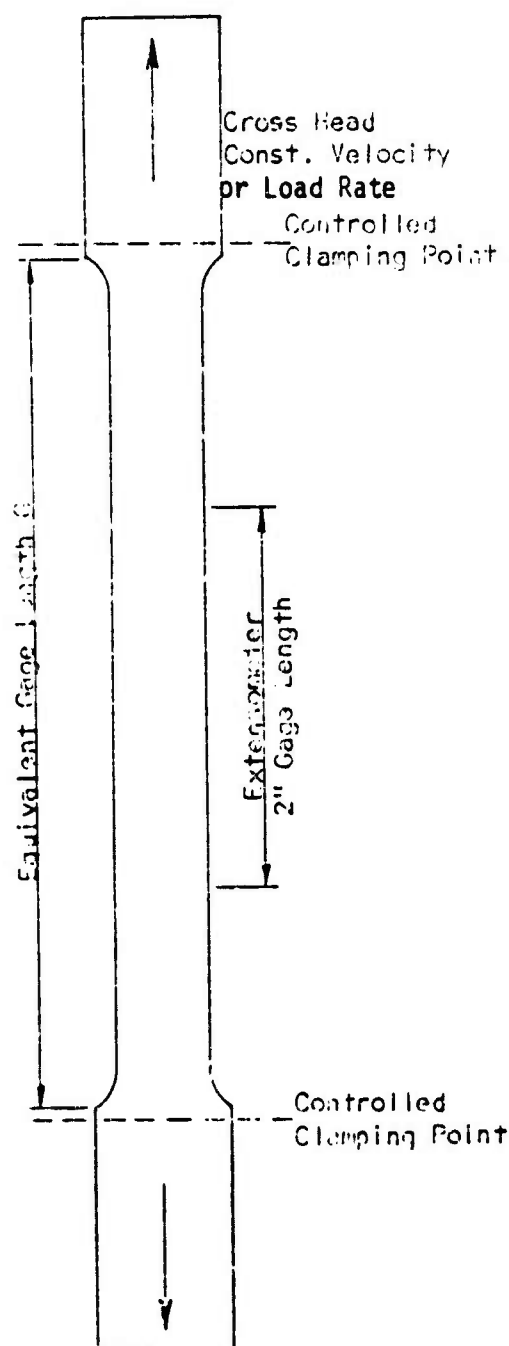


FIGURE 91. NEAT ADHESIVE SPECIMEN

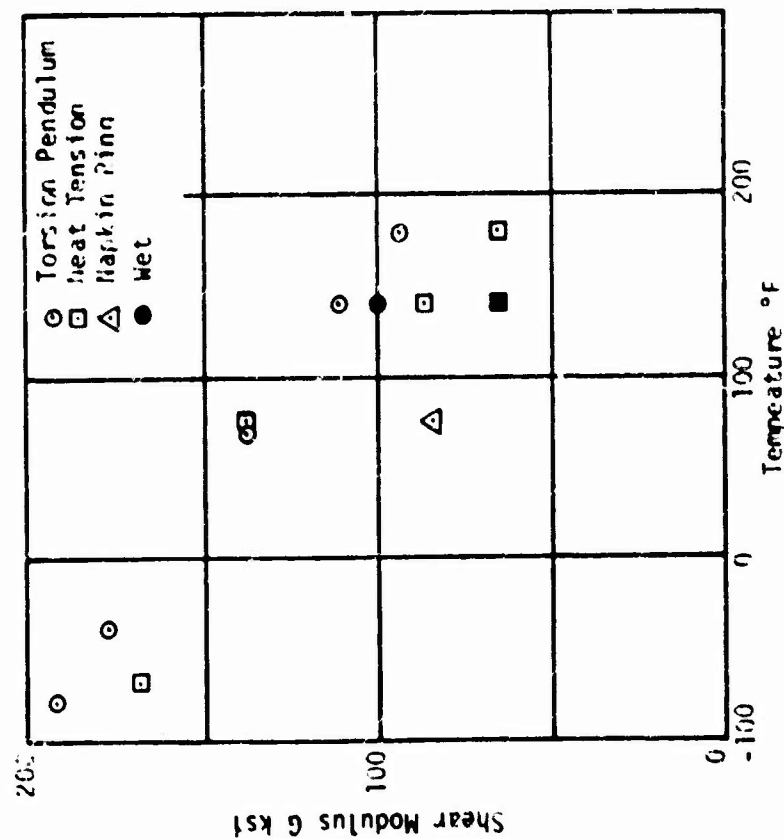


FIGURE 92. SHEAR MODULUS VS TEMPERATURE FM73

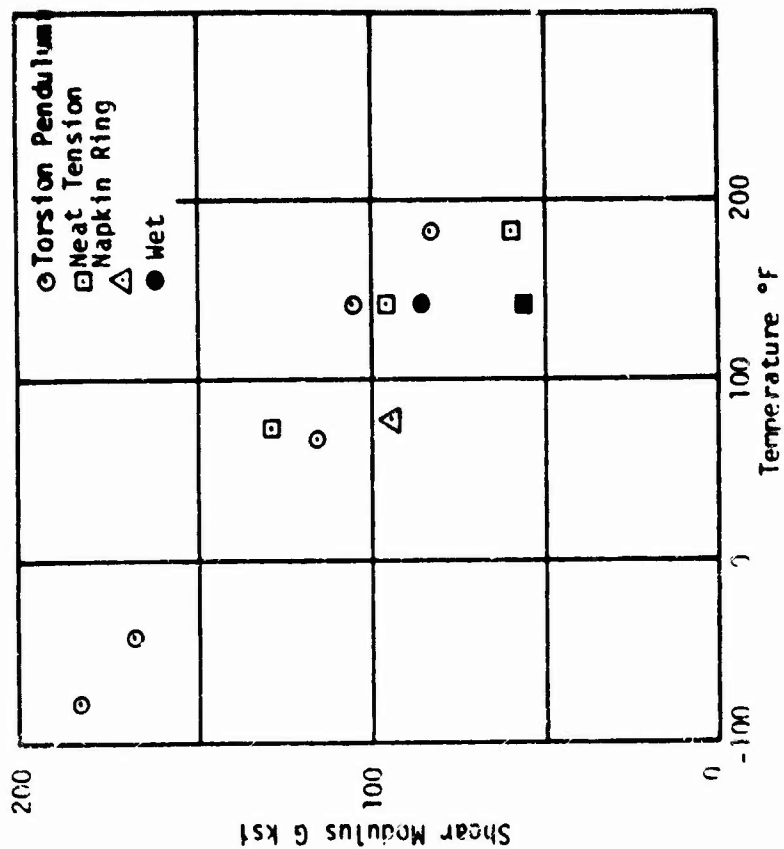


FIGURE 93. SHEAR MODULUS VS TEMPERATURE AF55

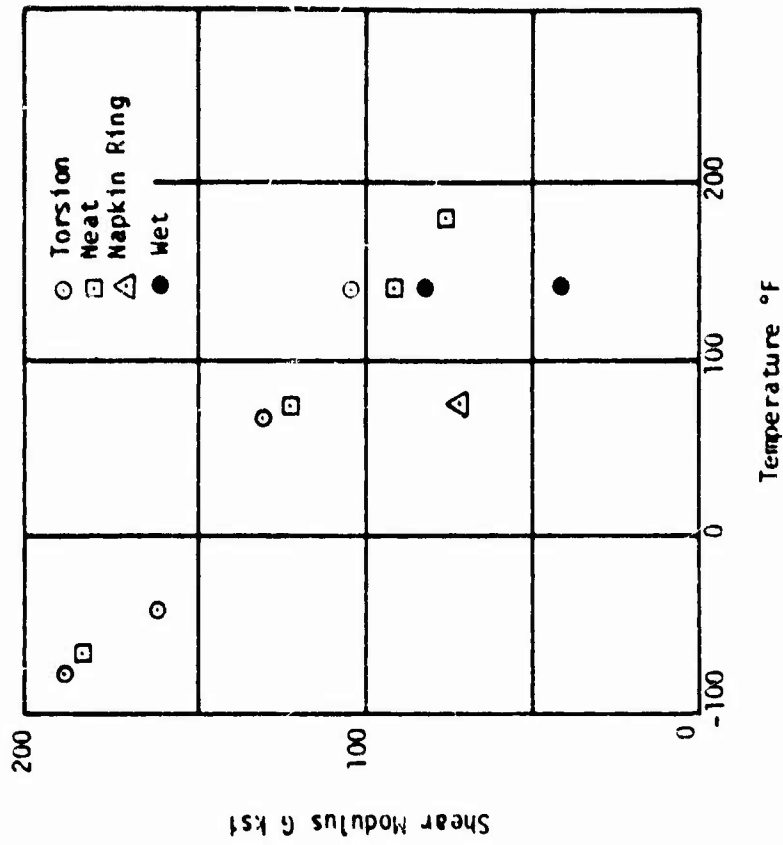


FIGURE 94 SHEAR MODULUS VS TEMPERATURE M1133

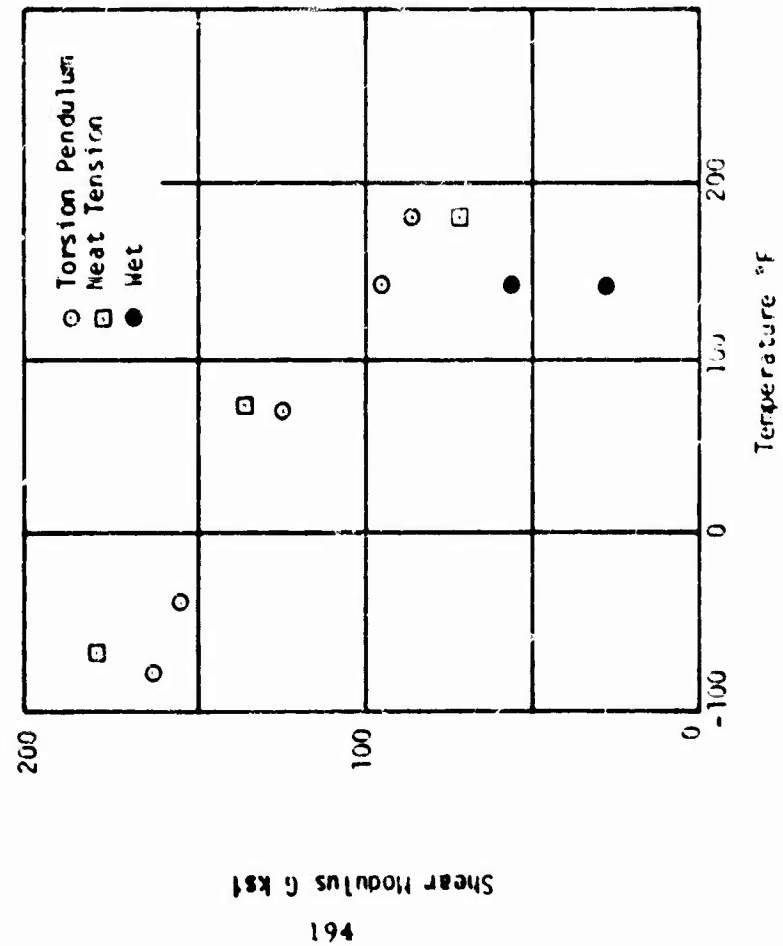


FIGURE 95. SHEAR MODULUS VS TEMPERATURE EA9628

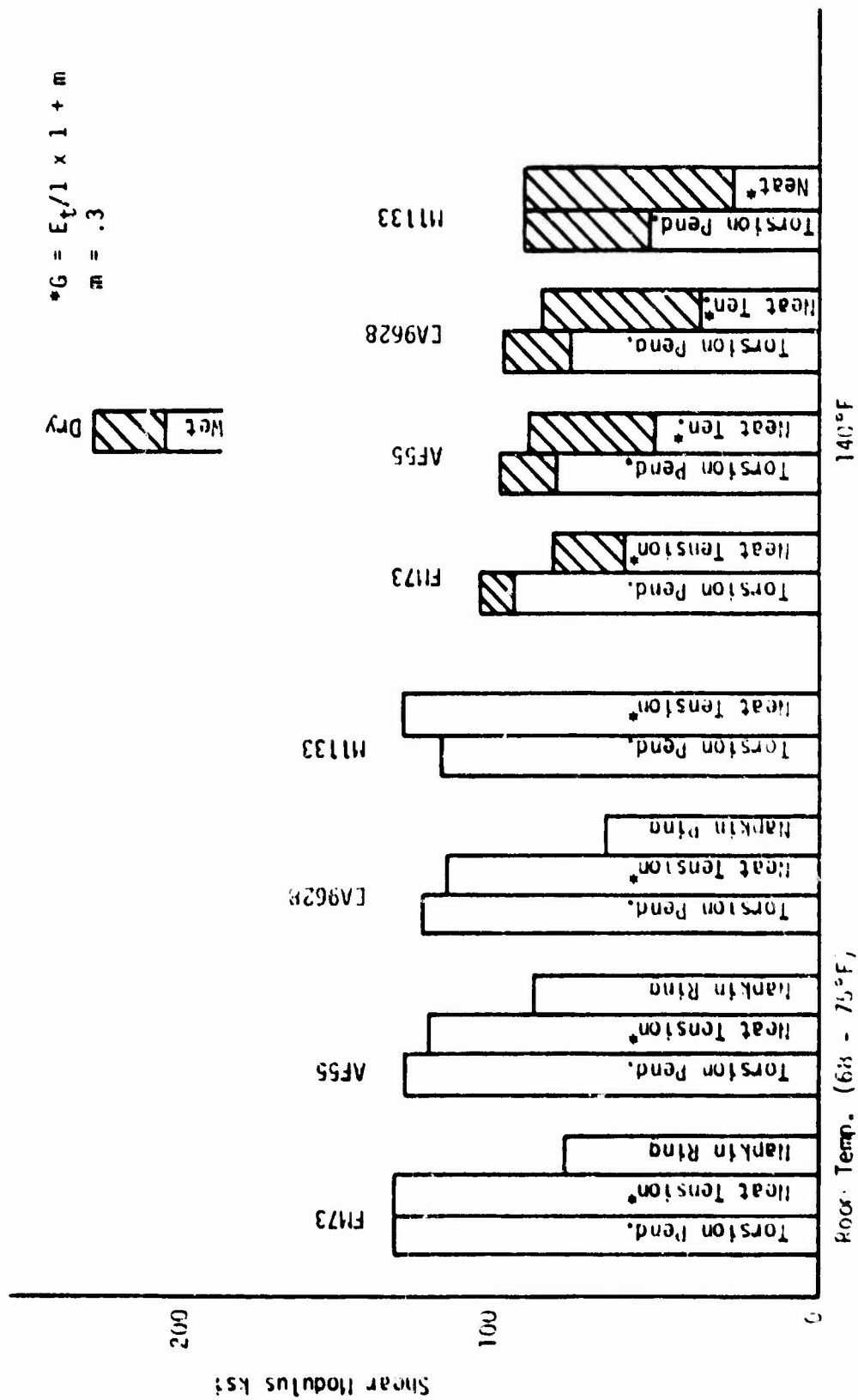


FIGURE 96. A COMPARISON OF SHEAR MODULUS FOR VARIOUS ADHESIVE SYSTEMS

6.3.1.9 Double Cantilever Beam

Double cantilever beam specimens of 7075-T6 bare alloy with the phosphoric acid anodize surface treatment were fabricated using the four adhesive systems. The test panels were bonded of .5 inch plate stock and the individual 1 x 14 inch specimens were machined from the bonded plate. The first test of this type in the program was a 120 day laboratory air exposure. The specimens were wedged open by torquing the two screws in the end of the specimen (see Figure 97) to an opening of approximately .20 inch. A measurement of the crack tip displacement was made, and subsequent measurements were made of the crack tip displacement over the exposure period. The strain energy release rate (G_I) was calculated from the following formula:

$$G_I = \frac{y^2 M h^2 [3(a + .6h)^2 + h^2]}{16[(a + .6h)^3 + ah^2]2}$$

Where:

G_I = Strain energy release rate, inch lbs/in²

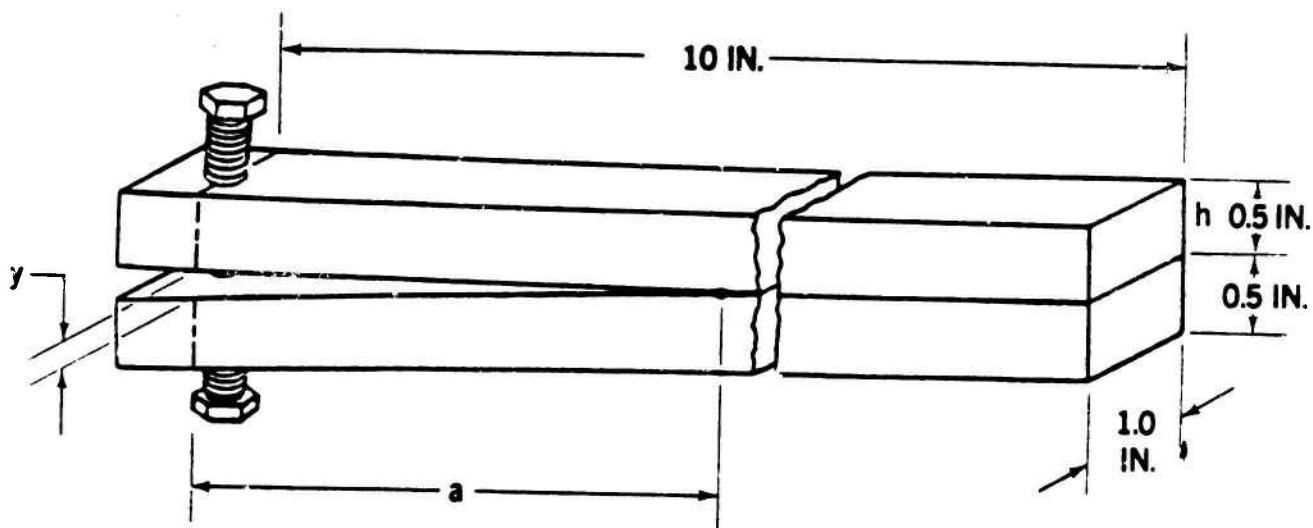
y = Displacement at the load point, inches

a = Distance from load point to crack tip, inches

h = Height of one beam, inches

E = Modulus of elasticity of adherends, lbs/in²

Besides the measurement for G_I , the mode of failure was recorded on all specimens. As is shown in the graph (see Figure 98), the G_I change, or crack growth, was very slow over the period of test, with little crack growth in the 120 day period. After the completion of these tests, the specimens were coated with an epoxy corrosion inhibiting primer system and placed at the beach exposure site. The crack extension will be measured on these specimens each month until they are comparable to the specimens exposed to 140°F and 95-100% RH, the testing of which is expressed elsewhere in this report. (See Section 6.4.1.6.)

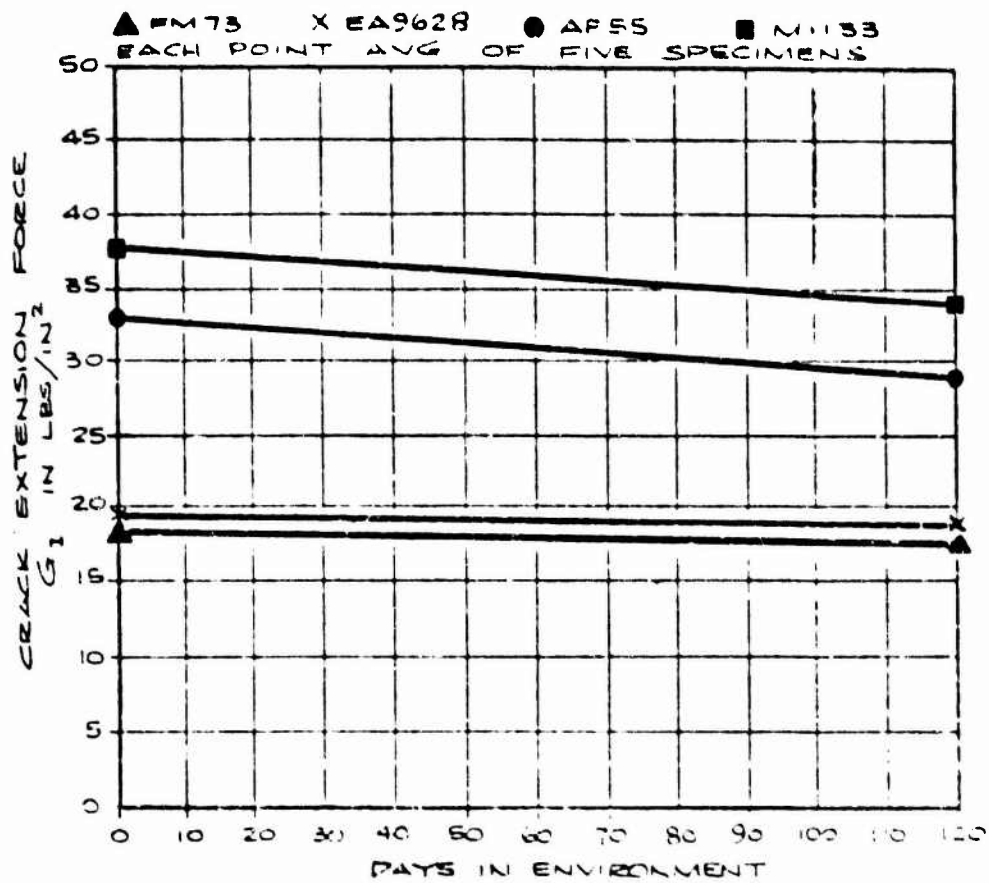


$$G_I = \frac{y^2 M h^3 [3(a + 0.6h)^2 + h^2]}{16 [(a + 0.6h)^3 + ah^2]^2}$$

WHERE:

- G_I = Strain Energy Release Rate - in.-lb/sq.in.
- y = Displacement at Load Point, Inches = 0.25 inch
- a = Distance from load point to crack tip, Inches
- h = Height of one Beam, Inches = 0.5 inch
- M = Modulus of Adherend, lb/sq in. = 10,300,000 psi

FIGURE 97. DOUBLE CANTILEVER BEAM TEST SPECIMEN



120 DAYS EXPOSURE AMBIENT TEMP.

FIGURE 98. DOUBLE CANTILEVER BEAM - AMBIENT EXPOSURE

6.4 Adhesives Environmental Resistance Tests

After the mechanical test program on the adhesives was started, the specimens for the environmental resistance tests were fabricated. For the mechanical tests, the phosphoric acid anodize was used as a standard surface treatment for the variable that would affect the investigation. The tests in the surface treatment portion of the program indicated an easy capability of producing, with FM73 adhesive, a bond that was resistant to the effects of 100% RH and 140°F. Other environments could affect the phosphoric acid anodize and would be tested with a comparison to the other surface treatments if this should be indicated in any test procedure.

Most of the test specimen configurations were similar to those used in the mechanical tests. Some specimen configurations were of designs that could be best used for environmental exposure comparison testing and had little or no value when tested in the static nonexposed condition.

Some of the test specimens were of a configuration that would allow exposure to some of the environment but not all of them. This is apparent with the RAAB type specimen (See Figure 99) that would be difficult to immerse in a test fluid and still be cycle stressed. A list of the types of specimens and the numbers used in each environment test are in Table 36.

The environments used in the exposures were normally general Military Specification requirements except in some cases the exposure time was extended to 120 days. The environment for bilge fluid was selected from a British report which had many bilge fluid analyses based on actual samples removed from aircraft in service (Section 8.9). The exposures in the various environment are detailed in Table 37.

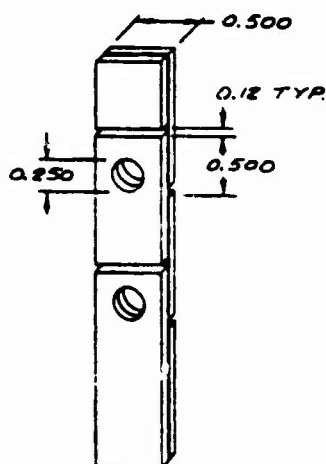


FIGURE 99. LAP SHEAR (RAAB) (TWO OF SEVEN TEST AREAS SHOWN)

TABLE 36
ENVIRONMENTAL RESISTANCE ADHESIVES

SPECIMEN TYPE	SPEC DWG	ALLOY	ALLOY THICK.	TEST AREA	ADHES	NUMBER OF SPECIMENS										REMARKS
						TT-S 735	JP 4	MIL-H 5606	140°F 100%	SALT SPRAY	CHEM MILL	BILGE FLUID	MIL-C 25769	IMMER CORR		
LAP SHEAR	FIG. 2.1	7075-T6 BARE	0.063	1 IN. x 0.50 IN.	FM73	5	5	5	5	5					35'	*SEE TABLE 2.4 SEE TASK 231 TOTAL 135 SPECIMENS
					EA9628	5	5	5	5	5						
					AF55	5	5	5	5	5						
					M1133	5	5	5	5	5						
STRESSED LAP SHEAR	FIG. 2.4	7075-T6 BARE	0.063	1 IN. x 0.50 IN.	FM73	5	5	5							TOTAL 60 SPECIMENS	
					EA9628	5	5	5								
					AF55	5	5	5								
					M1133	5	5	5								
T PEEL	FIG. 2.3	2024-T3 BARE	0.032	1 IN. x 9 IN.	FM73	5	5	5	5	5				15*	*SEE TASK 231 TOTAL 115 SPECIMENS	
					EA9628	5	5	5	5	5						
					AF55	5	5	5								
					M1133	5	5	5	5	5						
OPEN FACE CLIMBING DRUM	FIG. 2.5	7075-T6 BARE	0.020	3 IN. x 9 IN.	FM73	5	5	5	5						TOTAL 100 SPECIMENS	
					EA9628	5	5	5	5	5						
					AF55	5	5	5	5	5						
					M1133	5	5	5	5	5						
WEDGE CRACK	FIG. 2.11	7075-T6 BARE	0.125	1 IN. x 6 IN.	FM73	5	5	5	5*	5	5	5*	5*		*SEE TABLE 2.4 AND TASK 230 TOTAL 145 SPECIMENS	
					EA9628	5	5	5	5*	5	5	5*	5*			
					AF55	5	5	5	5*	5	5	5*	5*			
					M1133	5	5	5	5*	5	5	5*	5*			
DOUBLE CANTI-LEVER	FIG. 2.9	7075-T6 BARE	0.500	1 IN. x 14 IN.	FM73				5*						*SEE TABLE 2.4 AND TASK 209 TOTAL 20 SPECIMENS	
					EA9628				5*							
					AF55				5*							
					M1133				5*							
RAAR SUSTAINED LOAD	FIG. 2.10	7075-T6 BARE	0.063	0.200 SQ IN.	FM73				15						5 SPECIMENS FOR EACH ADHESIVE WILL BE TESTED FOR BASELINE DATA TOTAL 60 SPECIMENS	
					EA9628				15							
					AF55				15							
					M1133				15							
RAAB CYCLIC LOAD	FIG. 2.10	7075-T6 BARE	0.063	0.200 SQ IN.	FM73				15						5 TEST AREAS PER ADHESIVE AT 3 PEAK LOAD LEVELS TOTAL 60 SPECIMENS	
					EA9628				15							
					AF55				15							
					M1133				15							

TABLE 37
ENVIRONMENT TEST PARAMETERS

TEST ENVIRONMENT	EXPOSURE TEMPERATURE	EXPOSURE TIME SPAN	REMARKS
TT-S-735	AMBIENT	120 DAYS	IMMERSION
P-4	AMBIENT	120 DAYS	IMMERSION
MIL-H-6606	AMBIENT	120 DAYS	IMMERSION
RELATIVE HUMIDITY 95 TO 100 PERCENT	140°F	120 DAYS 30 DAYS (APPROX) ONE HR	METAL-TO-METAL HONEYCOMB WEDGE CRACK
SALT SPRAY 5 PERCENT	95°F	30 DAYS	
CHEM MILL ETCH	185° - 200°F	15 MINUTES DROP APPLICATION	PRIMER IMMERSION WEDGE CRACK
BILGE FLUID	AMBIENT	7 DAYS	WEDGE CRACK
ALKALINE CLEAN MIL C-257(9)	AMBIENT	7 DAYS	WEDGE CRACK
IMMERSION CORROSION 5 PERCENT NACL pH3 WITH ACETIC ACID	AMBIENT	30 DAYS	LAP SHEAR T-PEEL

6.4.1 Test Descriptions

6.4.1.1 Tensile Lap Shear

These specimens are the standard .500 inch over lap shear specimens bonded with .063 inch plates. They were not the blister shear type since some effect of adhesive flash was desirable to represent what can be considered normal on a bonded structure. The alloy of the adherends was 7075-T6 bare.

The exposures were in TT-S-735 Standard Test Fluids, Hydrocarbon Type I; MIL-H-5606 Hydraulic Fluid, Petroleum Base, Aircraft and Ordnance; and JP-4 Fuel. The immersion time was 120 days at room temperature. Additional specimens were exposed to salt spray (Federal Test Method Standard No. 151) and 140°F and 95-100% relative humidity. The results of these tests are shown in Figure 100. It can be seen that none of the test fluids had a large effect on the lap shear specimens.

The major loss in lap shear strength was for those specimens exposed in the unstressed condition to 140°F and 95-100% relative humidity for 120 days. The average loss in shear strength for FM73/BR127 adhesive was 18%; for the EA9628/9202 adhesive system the loss was 49%; for the AF55/EA3950 adhesive system, the loss was 54%; and the loss for H1133/6740 was 53%. This reduction in shear strength from exposure to an elevated temperature and a wet atmosphere indicated that moisture with heat was one of the primary criteria to be considered in the test programs.

6.4.1.2 Stressed Lap Shear

Lap shear specimens for the four adhesive systems were also stressed to 900 psi in creep test specimen fixtures and immersed in MIL-H-5606, TT-S-735 and JP-4 for 120 days. The residual shear strength indicated little or no effect from these solvents. The results of these tests are in Figure 101.

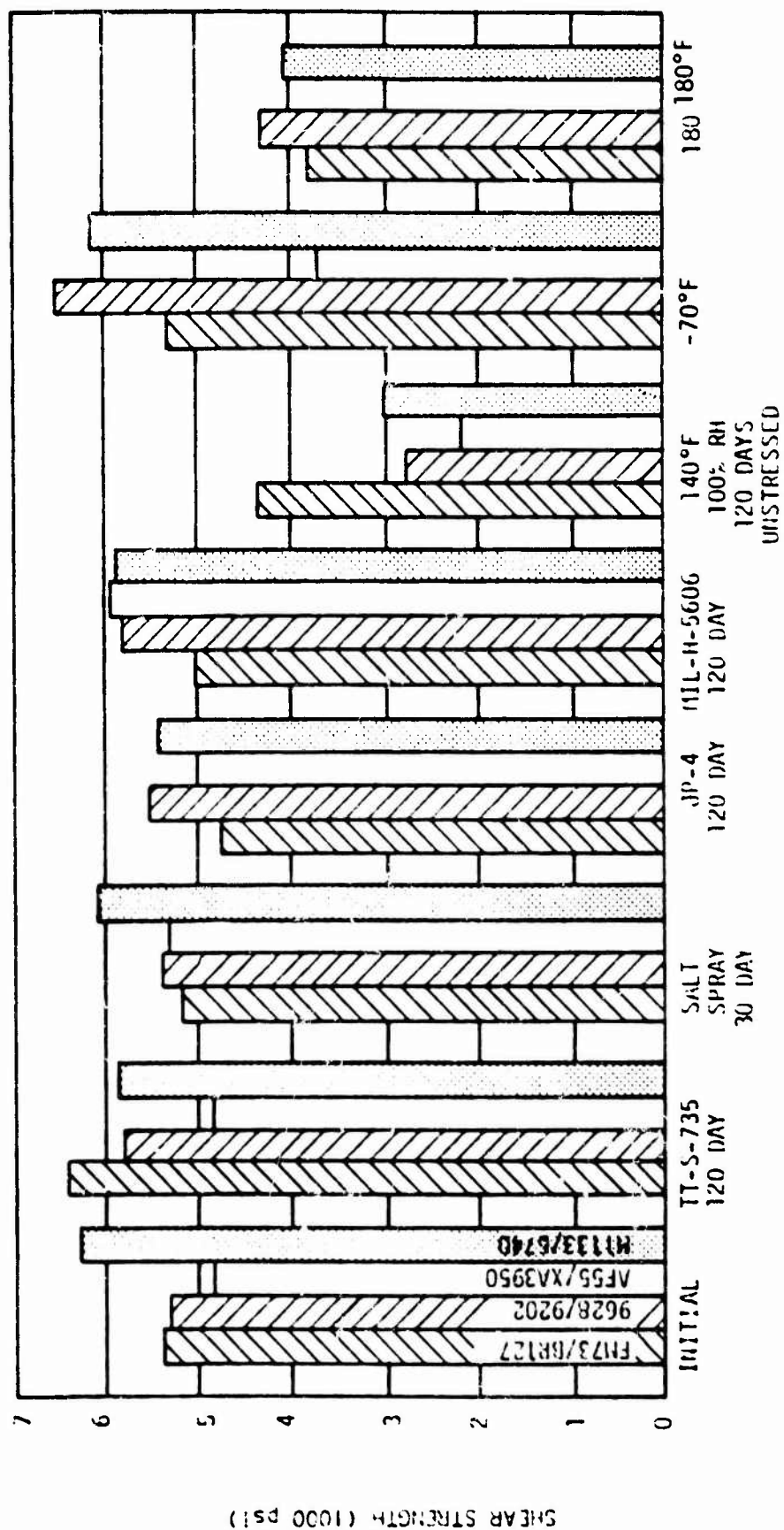


FIGURE 100. LAP SHEAR TEST

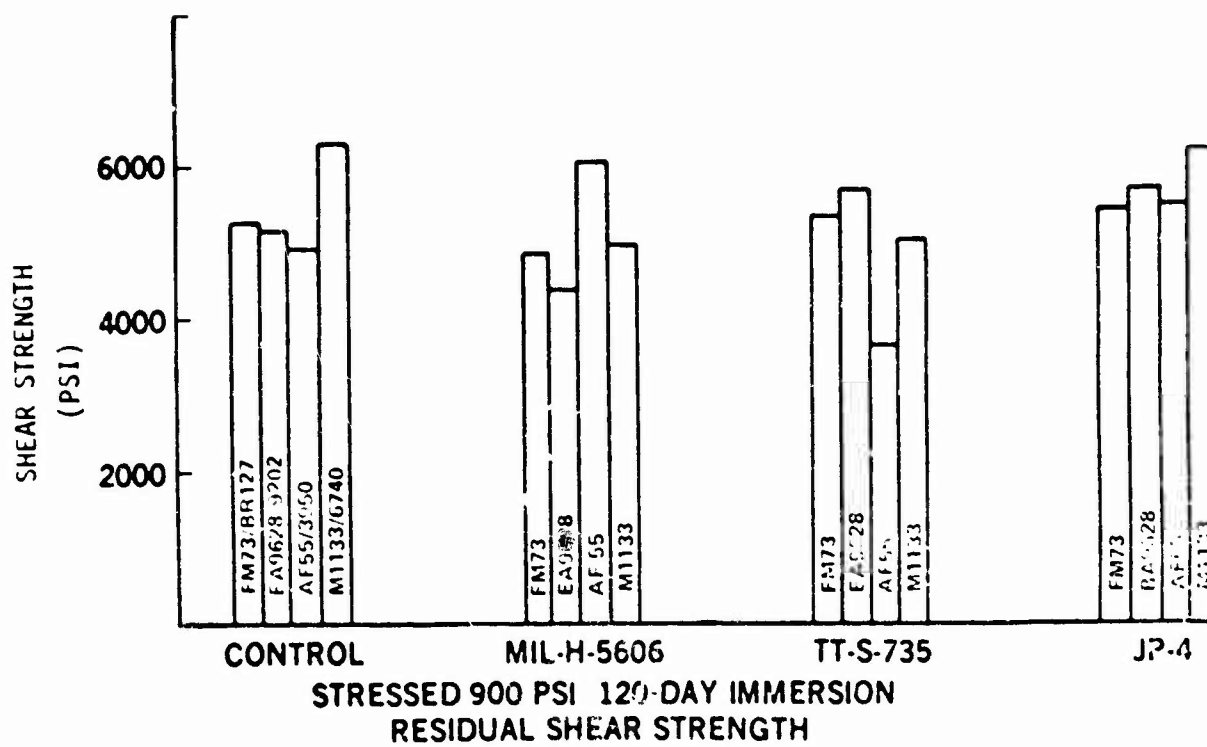


FIGURE 101. STRESSED LAP SHEAR

6.4.1.3 T-Peel Tests

T-peel tests were run on MIL-H-5606, TT-S-735 JP-4 and at elevated and subzero temperatures. One of the features of the adhesive that affects peel strength is the type of carrier used in the adhesive. As can be seen in Figure 102, the adhesive with a mat carrier had a much lower peel strength than those adhesives with the woven type of carrier. In two of the adhesives, the alternate type of carrier was also obtainable, these were FM73 and M1133. The room temperature peel strength of the FM73 with the woven carrier was over four times higher than the FM73 with the mat. The same condition can be seen in the M1133 adhesive. The effect of JP-4 on the adhesives was considerably greater on those with the woven carrier than on the adhesive with the mat. Since the mat carrier adhesive had a low peel with and without the JP-4 exposure, the effect of the JP-4 may not be discernible.

In the peel tests, the effect of 140°F and 95-100% relative humidity exposure was not as predominant as in the shear tests. The moisture, which reduces the shear modulus of the adhesive, does not substantially change the peel characteristics of the adhesive. Also, the specimen inherently has a larger cross section which extends the required exposure time to saturate the bond line with moisture.

The peel strength for the woven materials was reduced at the -70°F test temperature on EA9628 and AF55. The M1133 had a lesser effect. The adhesives with a mat carrier were affected by the -70°F test temperature in the case of FM73 but the M1133 did not have a reduction in strength. Since the aircraft will be exposed to minus temperature during most of the time that the fuselage will be stressed or under pressure, these peel numbers at -70°F are important considerations. Thirty-day salt spray had no effect on the peel strength of the adhesives.

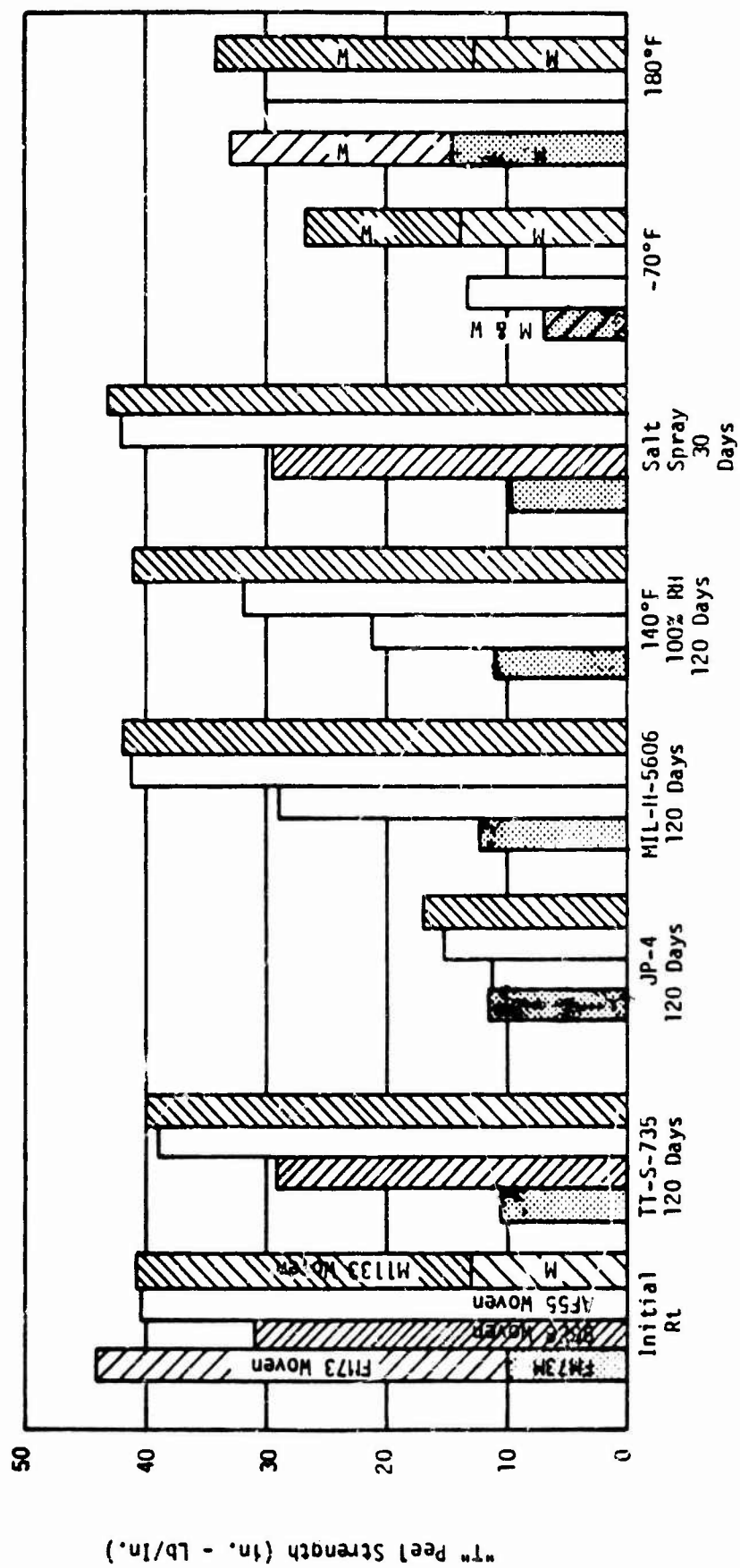


FIGURE 102. "T" PEEL TESTS

6.4.1.4 Open Faced Climbing Drum Peel

The open faced climbing drum peel specimen is used for determining environment effects on the adhesives. Its advantage is that the total bonding surface of the adhesive is exposed to the environment in contrast to a metal to metal bond where only the cut edge is exposed to the environment and the effect of the environment may not be apparent for a longer period of time. Also, the total adhesive surface exposure reduces the gradations of exposure that have to be inherent across a metal bond line. This test was used for moisture effect determinations before the advent of the wedge crack specimen.

The basic procedure is to bond only one skin to the honeycomb of a standard 3 x 12 inch honeycomb drum peel specimen. The specimen is then exposed to the environment for a specified time, the environment having access to the total adhesive surface through the honeycomb. The specimen is then removed from the environment and the second sandwich skin is bonded to the exposed honeycomb surface with a cold set fast cure two-part epoxy adhesive and the specimen is then peel tested.

Figure 103 shows minor effects from the environments. The FM73 with the mat carrier was consistently lower in peel strength as also was demonstrated in the metal to metal peel tests.

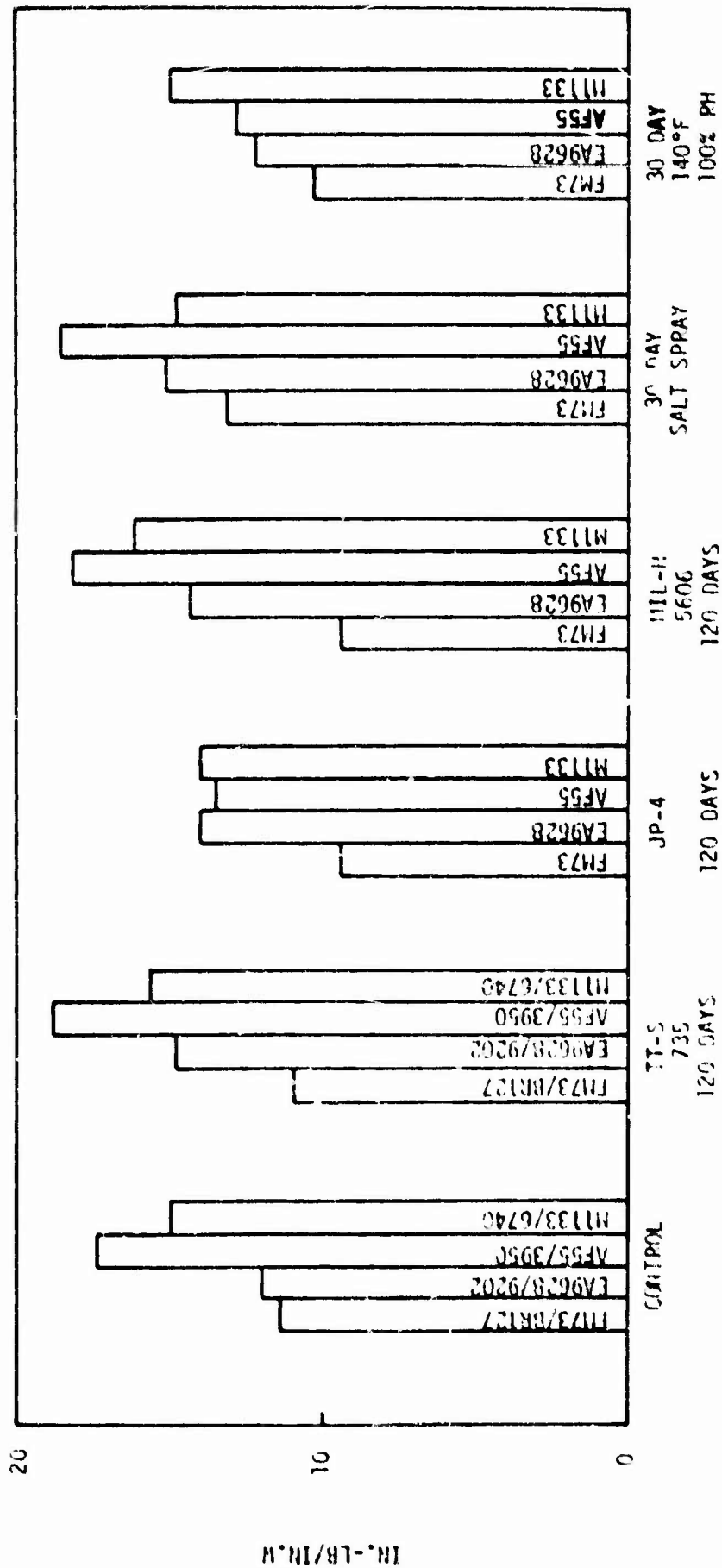


FIGURE 103. OPEN FACED CLIMBING DRUM PEEL

6.4.1.5 Wedge Crack Environment Tests

Wedge crack specimens were exposed to TT-S-735, JP-4, MIL-H-5606, salt spray and chem mill etch solution. The first three were immersion tests at ambient temperature for 120 days. The salt spray was for 30 days and the chem mill test was conducted by holding the wedge crack specimens in a vertical position with the wedged end up and dripping chem mill etch on the crack tip at 185 to 200°F for 15 minutes.

The first four tests were to determine service environment resistance, the chem mill test was to determine the resistance of the bond line to chem mill solution if chem milling of the bond assembly was required as a production operation.

In all of the tests, the failure mode of the wedge crack specimens was cohesive with crack growth averaging .25 inch or less, except in the case of AF-55 in JP-4 where the average crack growth was .46 inch.

The overall resistance of all the adhesives was very good in a test where the exposure is on the adhesive while a maximum tension stress is applied.

6.4.1.6 Double Cantilever Beam

Double cantilever beam specimens were fabricated, stressed, and exposed to 120 days of 140°F and 95-100% relative humidity. Crack growth measurements were taken weekly for the 120-day period (Table 38). A calculation for G_I , strain energy release rate in inch lbs/in², was made using the following equation:

$$G_I = \frac{y^2 M h^3 [3(a + .6H)^2 + h^2]}{16[(a + .6H)^3 + ah^2]^2}$$

Where:

G_I = Strain energy release rate, inch lbs/in²

y = Displacement at load point, inches

a = Distance from load point to crack tip, inches

b = Height of one beam, inches

M = Modulus of adherend, lbs/in²

TABLE 38
DOUBLE CANTILEVER BEAM

Adhesive System	Specimen Ident.	J	1	7	14	21	28	35	42	49	56	63	70	77	84	91	98	105	112	120	Failure Mode
FM73 / BR127	215-1-1	4.863	4.978	5.022	5.042	5.118	5.118	5.195	5.215	5.321	5.444	5.540	5.77	5.784	5.784	5.784	5.784	5.784	5.784	5.784	Cohesive
	-2	4.821	4.943	4.986	5.005	5.189	5.230	5.276	5.319	5.401	5.538	5.610	5.013	5.723	5.868	5.868	5.868	5.880	5.880	5.880	Bondline
	-3	5.060	5.291	5.357	5.369	5.500	5.607	5.692	5.895	6.013	6.094	6.244	6.205	6.311	6.457	6.457	6.531	6.531	6.531	6.549	Corrosion
	-4	4.970	5.234	5.320	5.354	5.631	5.780	5.842	5.892	5.994	6.123	6.223	6.231	6.249	6.338	6.338	6.431	6.458	6.458	6.530	At Edges
	-5	4.775	4.998	5.090	5.289	5.364	5.471	5.448	5.495	5.681	5.784	5.820	5.855	5.886	6.066	6.169	6.169	6.169	6.169	6.190	
EA9628 / EA9702	215-2-1	4.876	5.319	5.944	6.113	6.404	6.576	6.707	6.936	7.035	7.119	7.197	7.238	7.360	7.410	7.410	7.659	7.659	7.659	7.699	
	-2	4.932	5.289	5.864	5.996	6.255	6.402	6.526	6.569	6.658	6.716	6.822	6.933	7.255	7.288	7.300	7.351	7.405	7.405	7.473	
	-3	5.028	5.393	5.800	5.980	6.342	6.420	6.552	6.674	6.794	6.911	7.012	7.313	7.323	7.372	7.399	7.421	7.458	7.545	7.608	
	-4	5.100	5.436	5.996	6.024	6.507	6.704	6.751	6.912	7.013	7.125	7.191	7.290	7.390	7.512	7.512	7.676	7.850	7.820	7.984	
	-5	4.905	5.362	5.982	6.161	6.448	6.623	6.705	6.795	6.874	6.967	7.058	7.245	7.311	7.381	7.381	7.620	7.647	7.647	7.656	
AF55 / EA3500	215-3-1	4.561	5.539	5.963	6.182	6.597	6.726	6.815	6.838	6.914	7.011	7.093	7.173	7.219	7.328	7.422	8.284	8.284	8.361	9.064	
	-2	4.597	5.456	5.876	6.085	6.266	6.354	6.424	6.510	6.586	6.676	6.750	6.826	6.892	7.096	7.157	7.382	7.382	7.382	7.458	
	-3	4.467	5.431	5.810	5.995	6.263	6.413	6.509	6.595	6.733	6.828	6.871	6.886	6.900	6.926	6.989	7.319	7.360	7.360	7.769	
	-4	4.749	5.070	5.992	6.227	6.456	6.543	6.695	6.751	6.899	7.018	7.088	7.177	7.275	7.638	8.277	8.277	8.851	8.851	8.905	
	-5	4.565	5.504	5.999	6.208	6.343	6.514	6.718	6.795	6.868	7.082	7.209	7.429	7.893	8.709	8.793	8.793	8.878	8.878	9.276	
M1133 / H6740	215-4-1	4.709	5.265	5.791	6.178	6.423	6.592	6.723	7.001	7.192	7.334	7.426	7.477	7.580	7.700	7.750	7.817	7.912	7.946	8.095	
	-2	4.508	5.164	5.666	5.938	6.281	6.471	6.664	6.831	6.967	7.080	7.158	7.263	7.419	7.503	7.557	7.641	7.703	7.703	7.950	
	-3	4.558	5.236	5.802	6.030	6.355	6.587	6.745	6.993	7.156	7.311	7.444	7.546	7.628	7.802	7.897	7.924	7.970	8.076	8.148	
	-4	4.351	5.018	5.593	5.867	6.243	6.291	6.594	6.805	6.954	7.079	7.153	7.264	7.347	7.468	7.537	7.585	7.656	7.724	7.920	
	-5	4.285	5.023	5.477	5.807	6.292	6.356	6.493	6.763	6.915	7.083	7.220	7.330	7.371	7.507	7.580	7.626	7.709	7.770	7.988	

Crack growth record in inches 140°F at 95-100% RH

Figure 104 shows, graphically, the strain energy release rate decrease over the exposure period for each adhesive system.

It should be noted that the FM73 maintained a higher strain energy release rate over the period of test than the other adhesive systems. FM73 was also the only system that had the mat carrier; the other three systems used the woven carrier. The results of this test indicate the FM73 is much better at resisting a hostile environment under high stress than the other adhesives. This condition cannot at this time be attributed to the effect of the carrier or to the adhesives themselves. Further tests should be performed to make this determination.

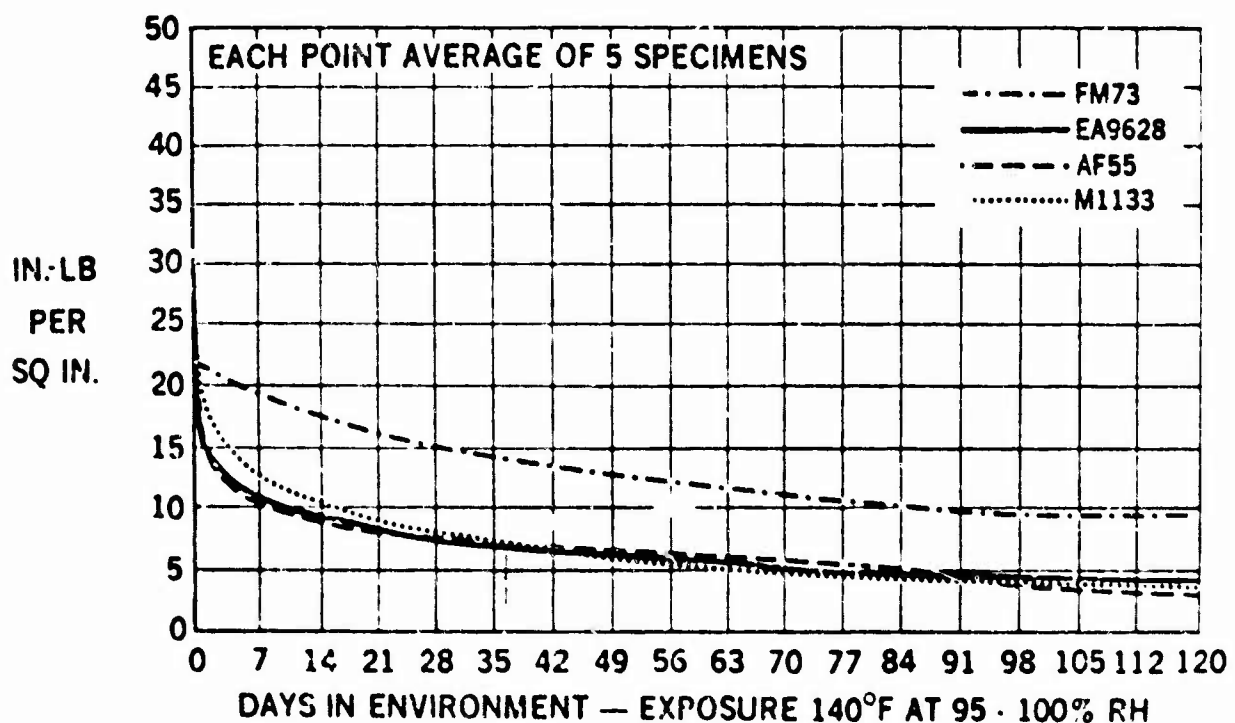


FIGURE 104. DOUBLE CANTILEVER BEAM TEST RESULTS

6.4.1.7 RAAB Sustained Load

The RAAB specimen is used for making comparison tests between adhesive systems in sustained and cyclic loads and in hostile environments. The specimen has two major advantages. The specimen has a .20 inch overlap. It also has 2.75 linear inches of exposed bond line. For environmental tests, this allows a rapid test procedure for determining the effects of any given environment. The maximum distance that the environment must penetrate the bond line is .125 inch to gain equilibrium. In addition, the specimen is configured where several may be tested at one time in one given test fixture. When one fails, it can be re-attached mechanically and the test continued on the other specimens in the string. For specimen configuration see Figure 99.

Some disadvantages exist, the primary being the difficulty in machining the specimens without overheating and damaging the test area.

The sustained test program included three load levels when the specimens were exposed to 140°F and 95-100% relative humidity. These load levels were 2050 psi, 1750 psi, and 1450 psi. Tests were conducted to failure. Figure 105 indicates that the FM73 resisted the environment and stress better than the other adhesives. Considering the results of the double cantilever beam specimens, this information is consistent. The spread between the woven carrier adhesives was much greater in the RAAB test than in the double cantilever beam test. EA9628 and AF-55 were relatively equal in their resistance to the environment, while M1133 demonstrated the poorest resistance in the tests performed.

6.4.1.8 RAAB Cycle Load

Cycle stressing of a bond joint in an environment can produce failures in a shorter period than when the specimens are loaded constantly in the same environment and under the same load. This was demonstrated by Frazier and Lajoi (AFML TR-74-26). Since the end product of a PABST program was to be a cycle loaded pressurized fuselage section, this phenomena became a primary part of the adhesive investigation.

The load cycle selected for the original part of this test program was one hour under load and 15 minutes with no load. The load maximums

were 300 psi, 900 psi and 1500 psi. This load cycle allowed the cycles for cycled stressed specimens to equal the number of hours under load for the constant sustained loaded specimens. Comparing the two charts, Figure 105 and 106 (sustained load and cyclic load) it becomes obvious that for the same environment and time, the cycled specimens were able to maintain approximately 60% of the stress of the constant stressed specimens to failure. This cycle effect, along with the cycle rate, cycled environment and its effect became a major project in the 600 series of the PABST program.

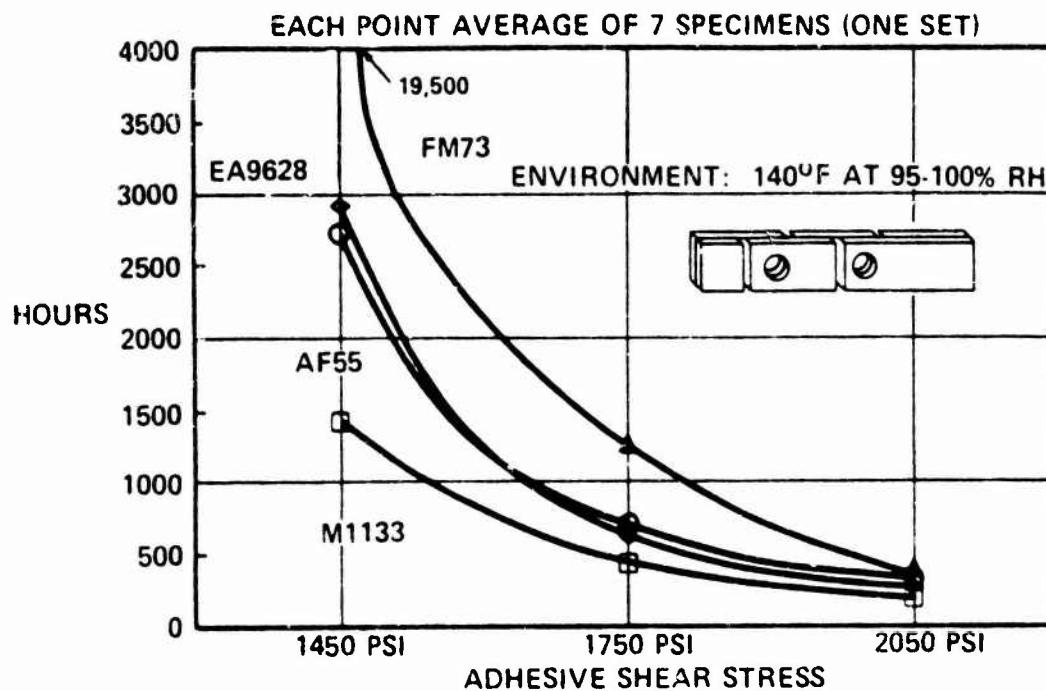


FIGURE 105. RAAB SUSTAINED LOAD

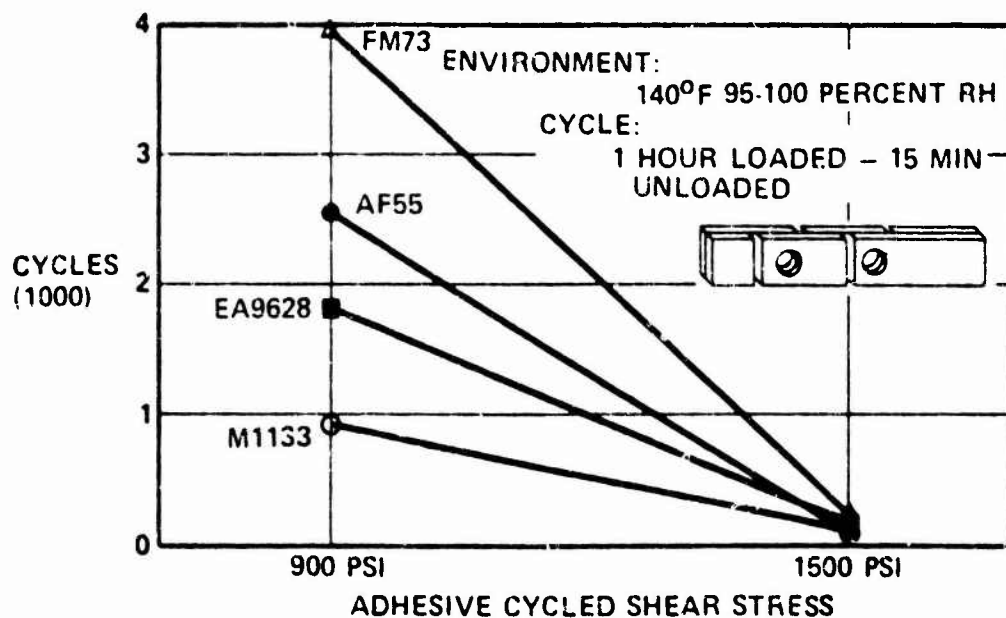


FIGURE 106. CYCLED LOAD/ENVIRONMENT TEST

SECTION VII

PROCESSING TOLERANCE ALLOWABLES

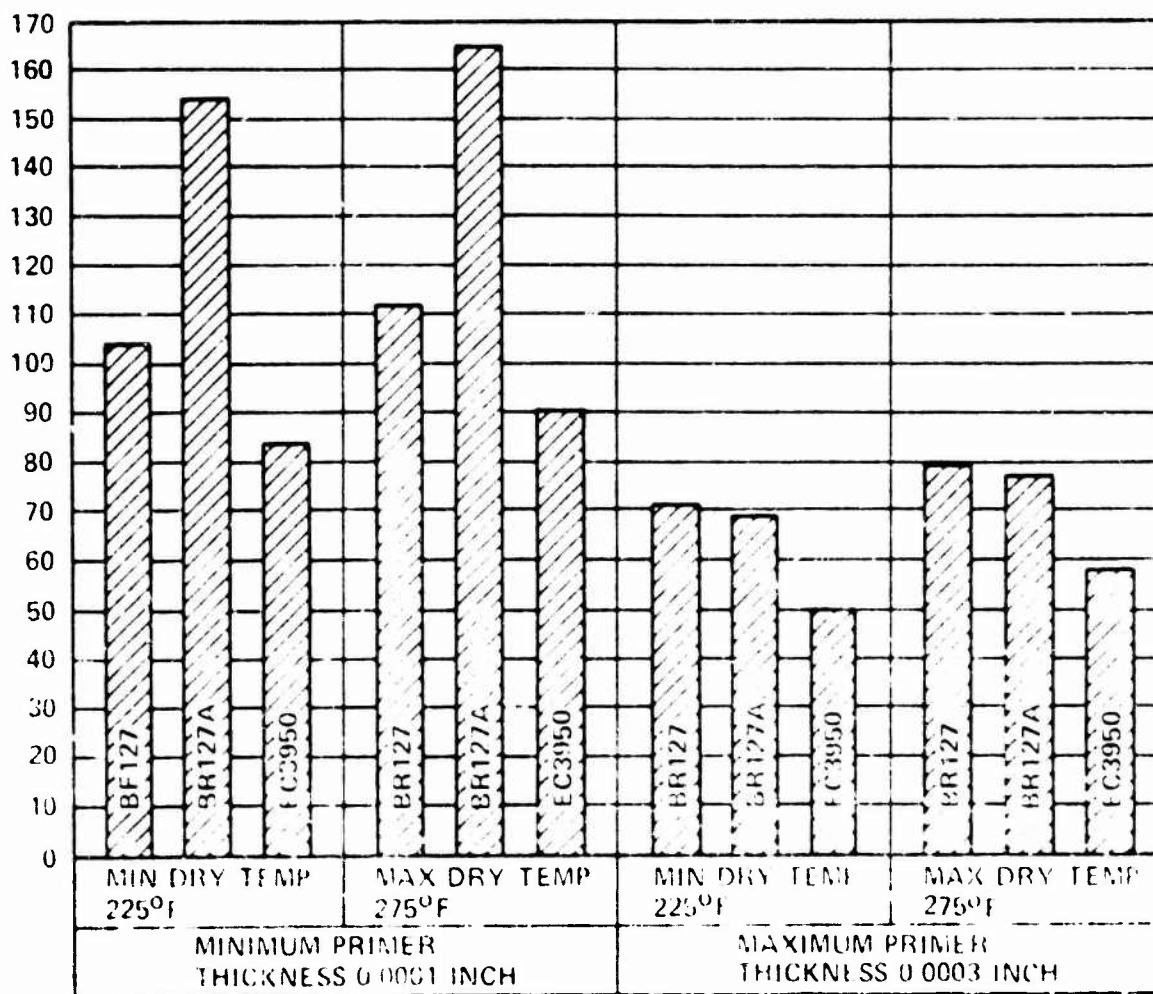
7. Introduction

In the normal operation of a production facility, it can be expected that daily variations will span the total allowable tolerances tested in the mechanical test part of this program and will in some instances be outside these tolerances. This part of the test program was conducted to determine the system characteristics when the processing exceeds the normal tolerance. This information will also assist the Liaison Engineer in his decision making when presented with a rejection tag.

7.1 Primer Processing Variables

The two major variables that are associated with primer application and cure are the applied primer thickness and variations in cure temperature. The applied primer thickness determinations have two problems that can allow wide variations. The application of the primer in the production area relies on the color of the applied primer as a gauge in determining when the proper thickness has been obtained. Unfortunately, most of the color in many of these corrosion inhibiting primers comes from the corrosion inhibitor itself. The corrosion inhibitors have a high settle rate in the primer, and constant agitation is required to maintain a uniform, evenly distributed mixture. If the material is not properly mixed and maintained in that condition during the priming operation, the operator can, inadvertently, apply too much primer while trying to obtain the proper color level on the part. The second problem is the difficulty of measuring the primer thickness instrumentally at the low end of the tolerance. The primer thickness tests were done at two levels, the minimum and maximum manufacturers recommendation. This testing was subsequently expanded to thicker levels since, in production, minor variations in the primer agitation during application could permit the painter to effectively apply more material than desirable. The primary effect of thick primer is a reduction in peel strength. Since peel strength is of primary importance on a pressurized fuselage at the frame tee to skin joints, very careful control is desirable. Primer that is too thin does not affect the mechanical properties but does reduce the environment resistance of the system in nonbond joints. Figure 107 shows the average peel strengths of BR127, BR127A and EC3950,

METAL TO METAL CLIMBING DRUM
POUNDS PER INCH WIDTH (PIW)



TEST TEMP 70°F AVG OF FIVE SPECIMENS

FIGURE 107. ADHESIVE PRIMER VARIABLES

the three primary candidate primer systems, at the two levels of thickness and two levels of dry temperature, when tested at -70°F. A drop-off in peel strength can be seen in the -70°F temperature tests at the maximum primer tolerance. Figure 108 shows the drop-off when primer thickness exceeds the maximum tolerance.

The two levels of primer dry (or cure) were the manufacturers recommended cure plus and minus 25°F. This spread of tolerance can be easily controlled in production and all cures are expected to fall within this tolerance. Figure 107 indicates that very little affect was noted by this variation in primer cure.

Lap shear results are in Figure 109. The wedge crack specimens had 100% cohesive mode of failure. Salt spray tests are in Figure 110.

All of the test specimens in Figures 107 and 109 were bonded with M1133 adhesive to reduce the effect of variables caused by the use of different adhesives and because M1133 has one of the highest peel strengths of the four adhesives in the test program.

It is apparent that, for a given design, the controls on primer application may be a critical part of the production process.

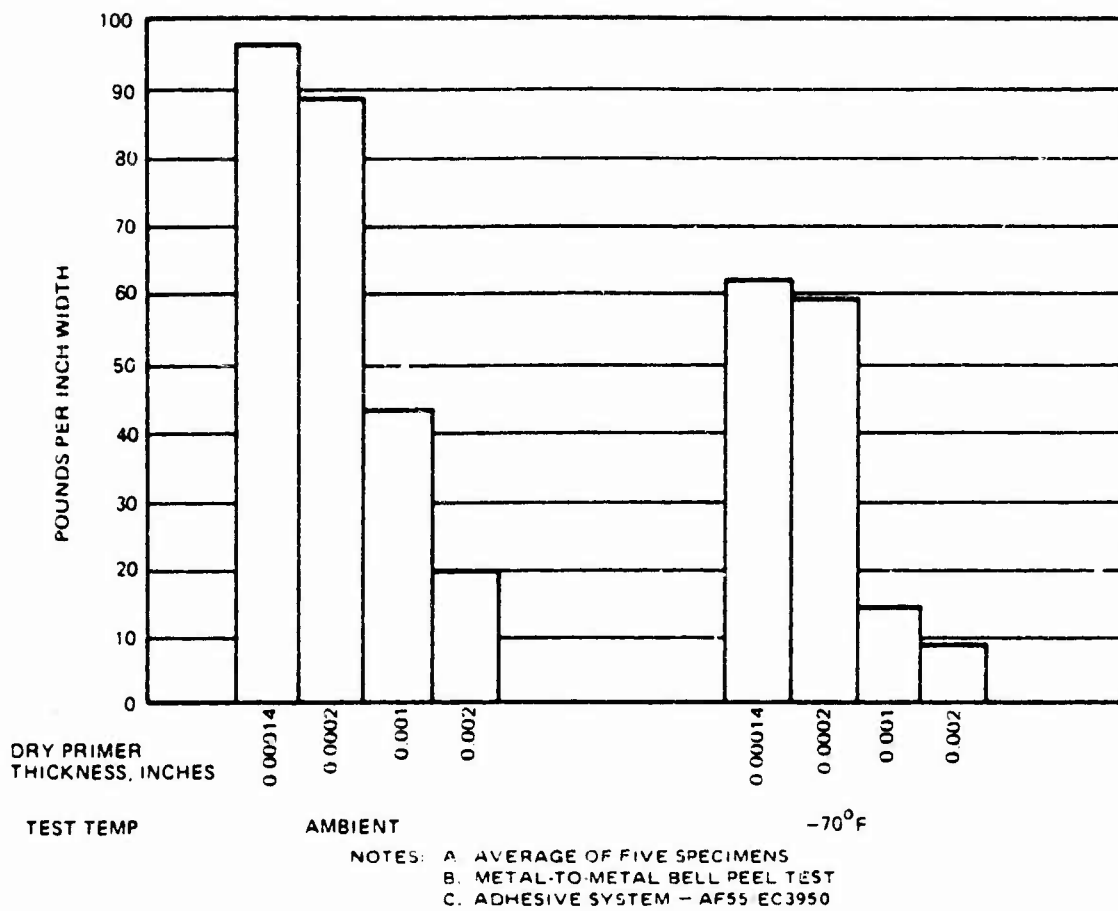


FIGURE 108. ADHESIVE PRIMER VARIABLES

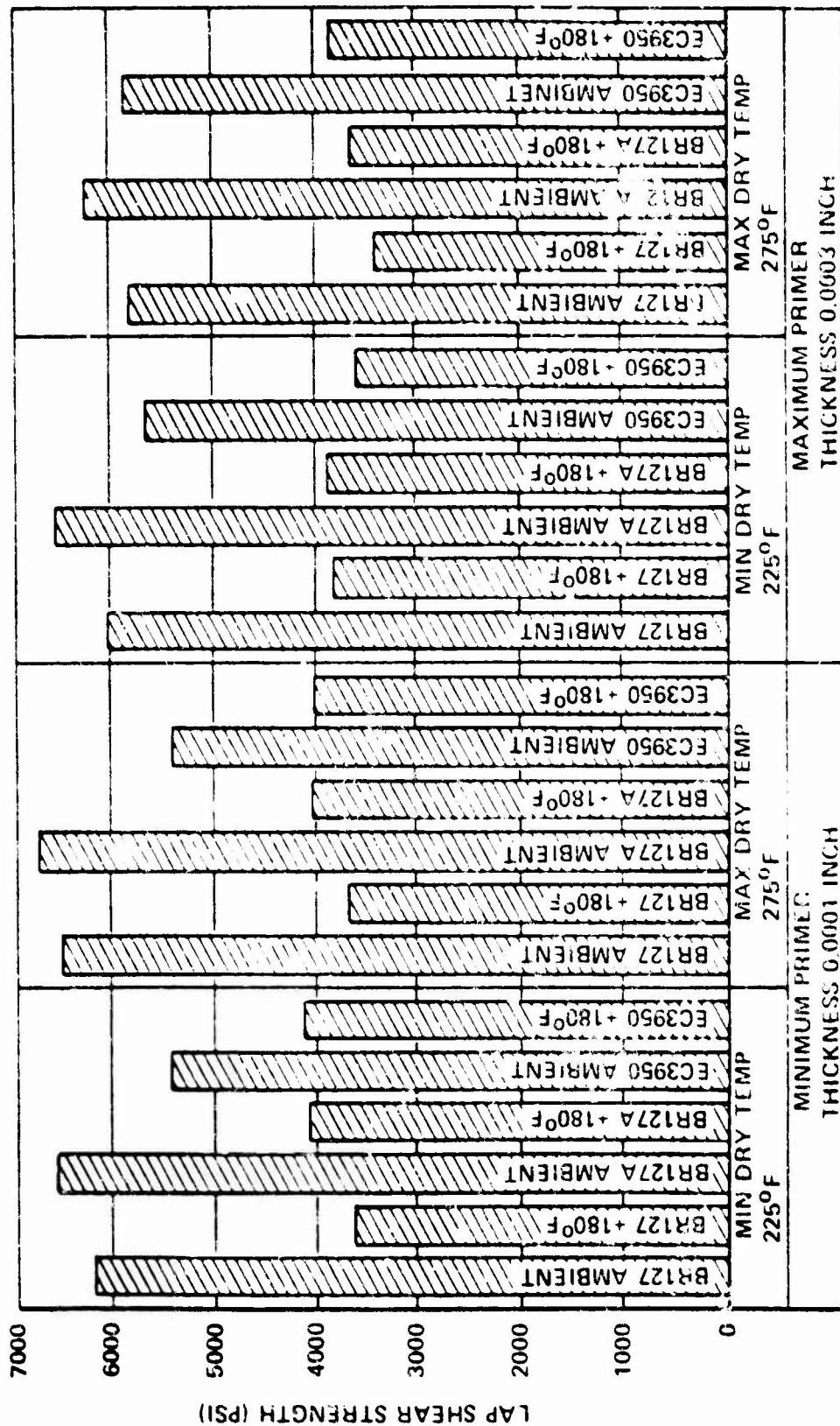
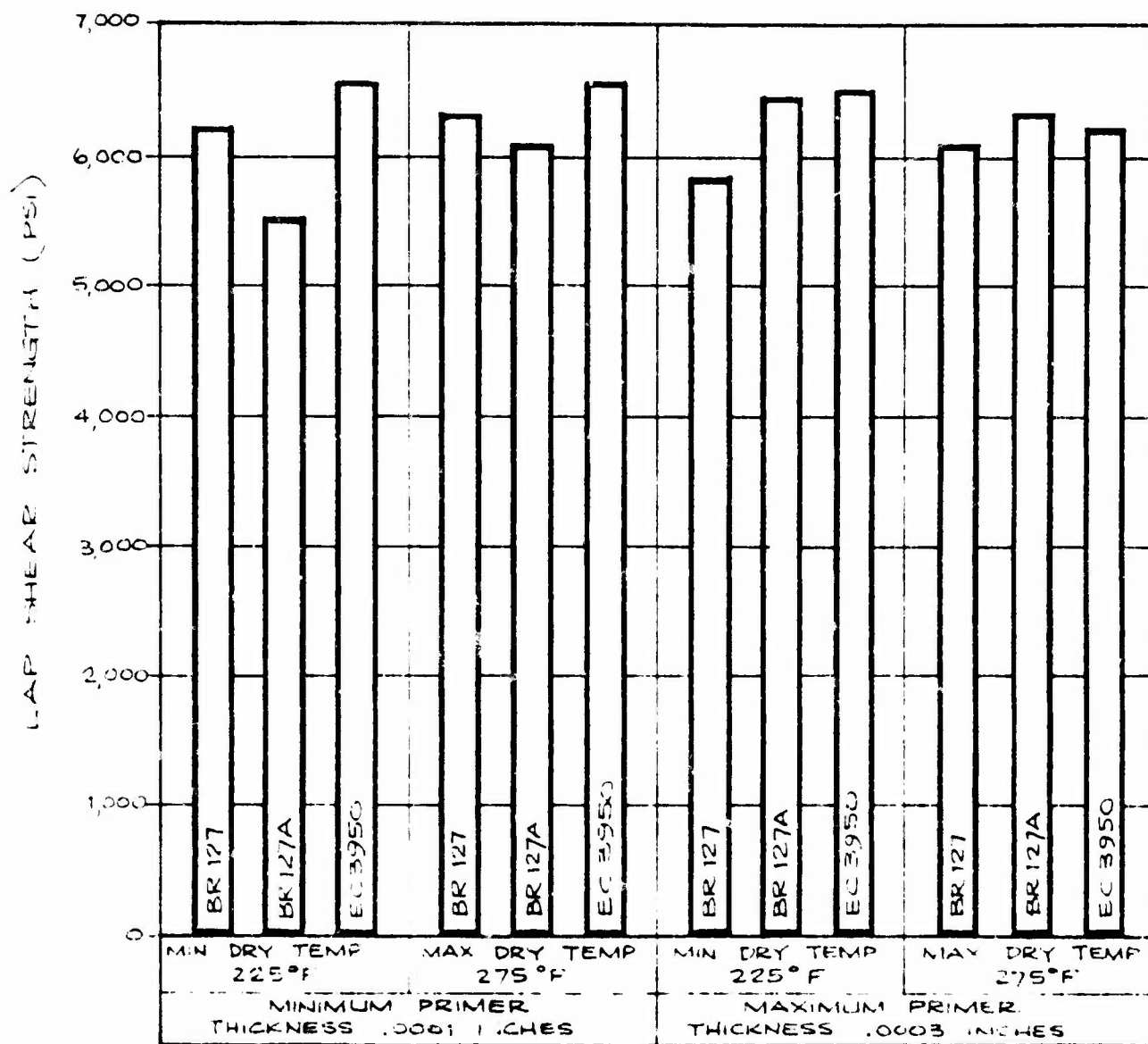


FIGURE 109. ADHESIVE PRIMER VARIABLES



NOTE: 30 day salt spray exposure

FIGURE 110. ADHESIVE PRIMER VARIABLES

7.2 Adhesive Processing Variables

Adhesive processing variables considered on the four candidate adhesive systems were heat-up rate during cure, and adhesive out time. The heat-up rate during cure test was to indicate if any of the systems were sensitive to a rapid or slow temperature rise. Some adhesives have a tendency to become porous during a rapid temperature rise while others are not affected. Conversely, some adhesives will gel during a slow temperature rise to cure and not properly flow and wet the adherend. This condition is clearly evident on some of the nylon epoxy types of adhesives.

The adhesive out time tests were to determine if the adhesives would be stable and not "c" stage too quickly at "clean room" temperatures and humidity levels. A certain amount of stability and resistance to moisture absorption is required to permit manufacturing time to lay up, bag and cure large complicated assemblies. The heat-up rate tests were conducted at two levels; 15-20 minutes from ambient to cure temperature (250°F) and 120 minutes from ambient to cure. These times represent rates that can be expected in production on thin structure and large mass structural sections.

In the adhesive out time tests, samples of each adhesive was exposed to the normal clean room environment for periods of one week, one month and two months. The plastic separator sheet was left in place on the exposed samples.

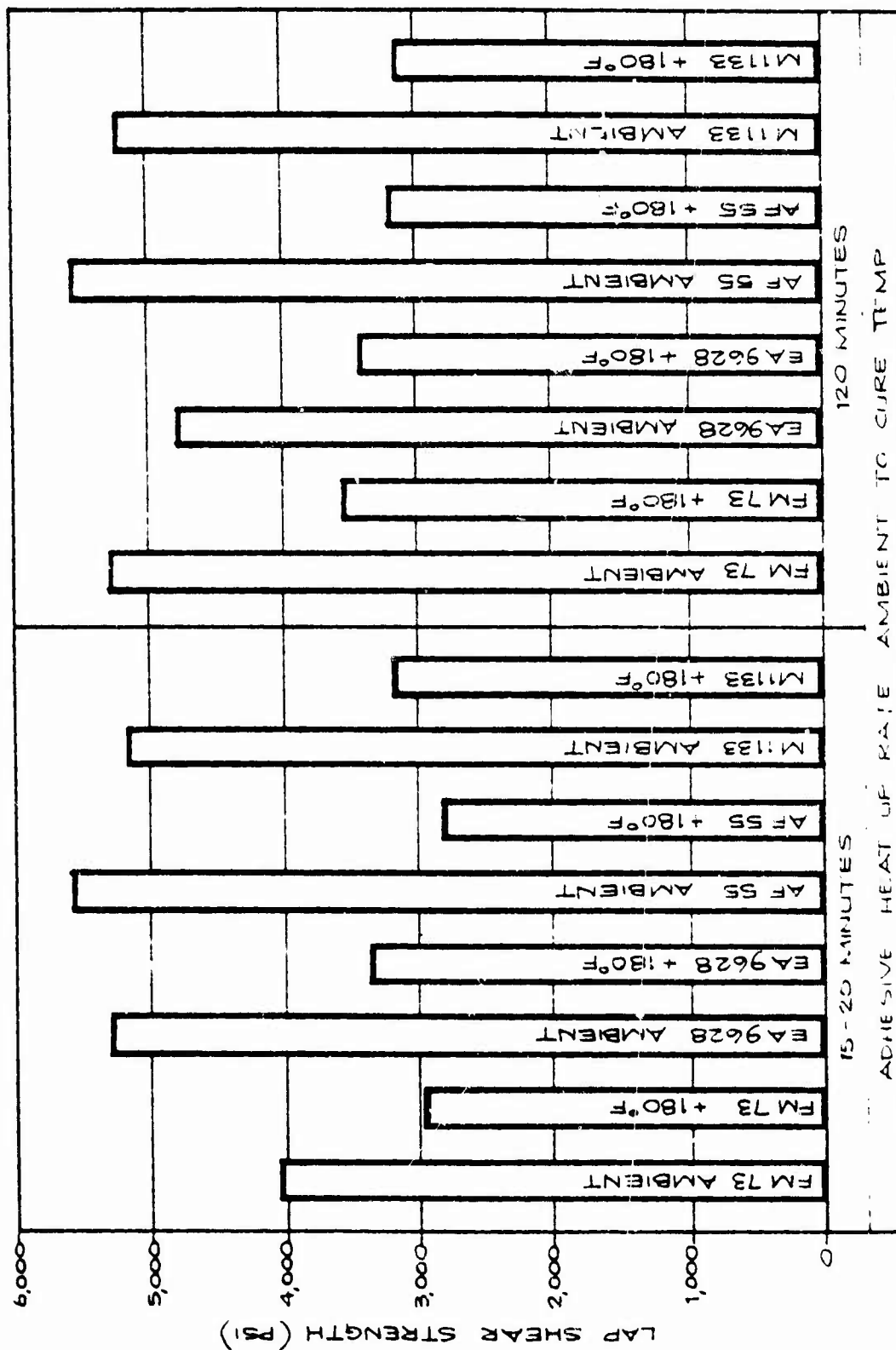
Specimens consisted of wedge crack coupons exposed to an environment of 140°F at 95-100% relative humidity for a period of one hour and tensile lap shear coupons tested at room temperature and 180°F after a 10 minute soak at temperature.

Results of tests showed that the FM73/BR127 adhesive system was sensitive to the heat-up rate of the cure cycle. It was noted that a porous glue line was obtained on the lap shear coupons at the high rate of heat-up with a strength drop of 900-1100 psi below the specimens cured at the slower heat-up rate. Results of out time exposure show no significant loss of mechanical properties on the FM73 in the tests performed. Handling properties of the FM73 adhesive film remained excellent.

EA9628/EA9202 did not appear to be affected significantly by the heat-up to cure rate. Results of out time exposure on both lap shear and wedge crack specimens show little change in values. The handling properties of the material changed in that the material became very dry and brittle making it difficult to work with.

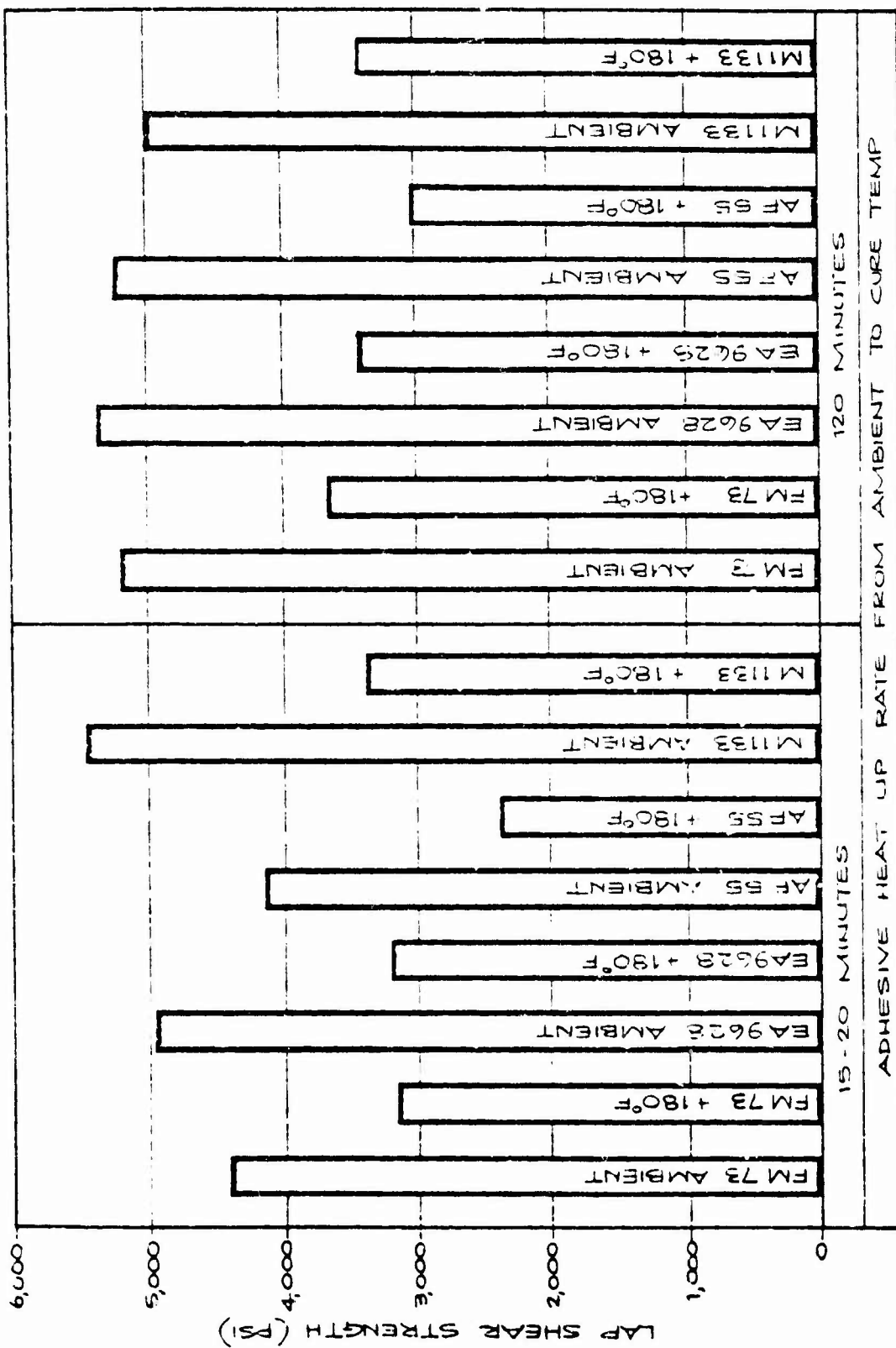
AF55/XA3950 adhesive system appeared to be affected by the heat-up rate of the cure cycle after exposure of the adhesive film to the room temperature environment exceeded 7 days. Lap shear values show a strength loss of approximately 1,000 psi after 30 days exposure and a fast heat-up rate during the cure cycle. Bond lines also exhibited porosity. Wedge crack coupons showed an increase in the initial crack length prior to test as the material exhibited lower peel characteristics. Handling properties of the material became very poor after 30-day exposure time, all initial tackiness was lost and the material became very dry and brittle.

M1133/M6740 adhesive system showed a strength loss of approximately 1,000 psi at the high heat-up rate after 60 days exposure of the adhesive film to the room temperature environment. Also a porous glue line was exhibited. Wedge crack environmental properties were not affected and the handling properties of the material remained good. Results of adhesive variables tests are in Figures 111, 112 and 113.

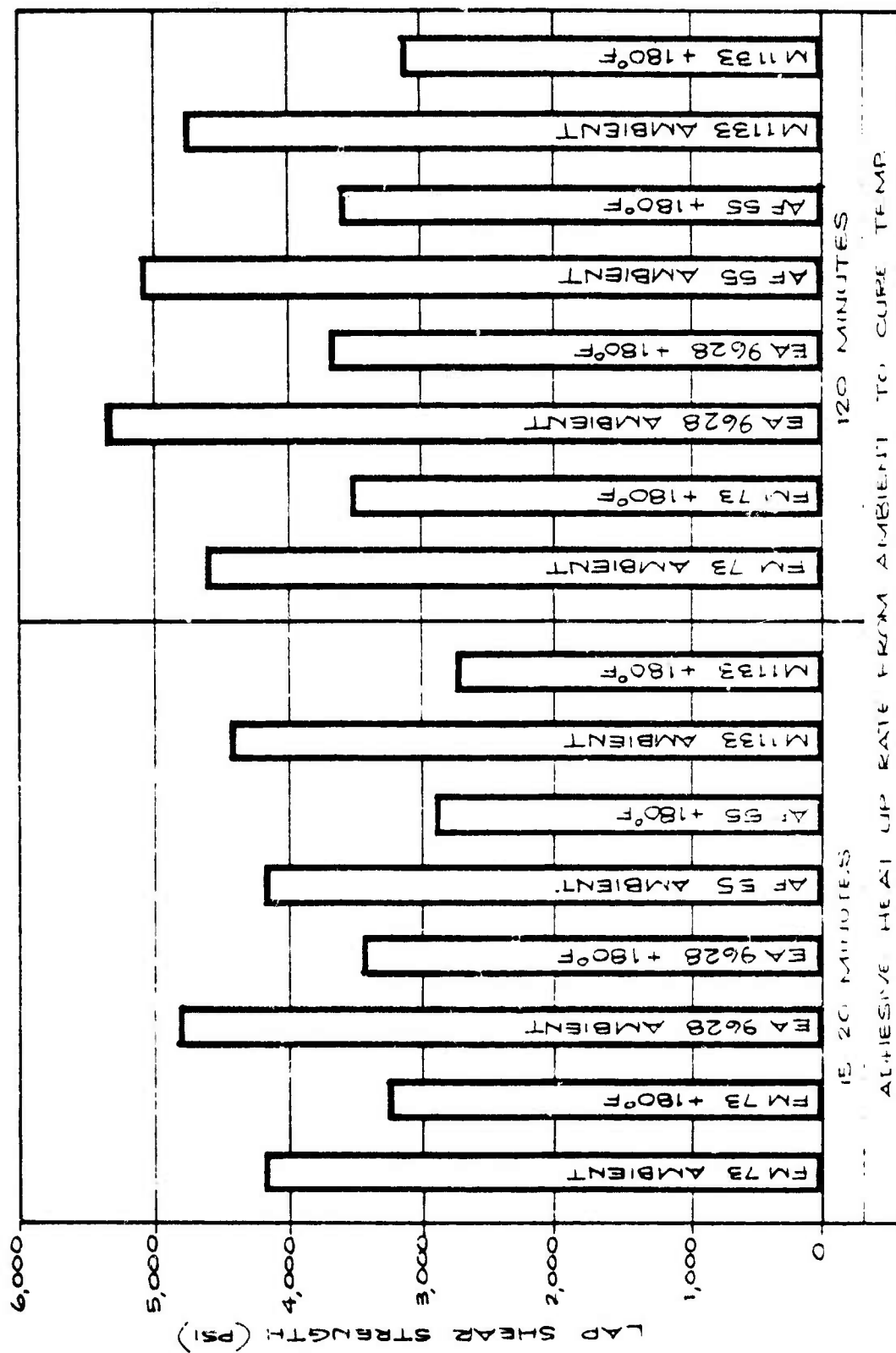


NOTE: Tensile Lap Shear 7 Day Ambient Exposure

FIGURE 111. ADHESIVE VARIABLES



NOTE: Tensile Lap Shear 30 Day Ambient Exposure
FIGURE 112. ADHESIVE VARIABLES



NOTE: Tensile Lap Shear 60 Day Ambient Exposure
FIGURE 113. ADHESIVE VARIABLES

7.3 Adhesive Batch Tests

These tests were performed to determine the consistency of the FM73 adhesive system and also the compatibility of different primer batches with different adhesive batches. The types of tests were lap shear tested at room temperature and wedge crack exposed to 140°F and 95-100% relative humidity. All the results of the wedge crack tests were cohesive mode of failure. The lap shear tests were very consistent indicating compatibility and consistency from batch to batch. The lap shear test results are in Table 39.

7.4 Instrumental Analysis of Adhesive

An instrumental analysis of FM73 was performed by McDonnell Aircraft of St. Louis. The results of the program are considered company sensitive. The procedures followed are diagramed in Figures 114 and 115.

TABLE 39

ADHESIVE BATCH TESTS

Spec. Ident	Adhesive Primer Batch 651 Adhesive Film		Adhesive Film Batch 125 Adhesive Primer	
	Batch 121	Batch 125	Batch 127	Batch 659
220-1	6310 psi	5850 psi	6080 psi	6480 psi
-2	6291 psi	5811 psi	6060 psi	6360 psi
-3	6427 psi	5981 psi	6200 psi	5760 psi
-4	6543 psi	6150 psi	6780 psi	6480 psi
-5	6350 psi	6113 psi	5980 psi	5960 psi
	6384 avg	5981 avg	6220 avg	6208 avg
				5912 avg
				6212 avg.

NOTE: All failures cohesive

Tensile Lap Shear Strength

Adhesive System: FM73 Adhesive Film /BR127 Primer

Surface Preparation: Phosphoric Acid Anodize

Metal Adherends - 7075-T6 Nonclad

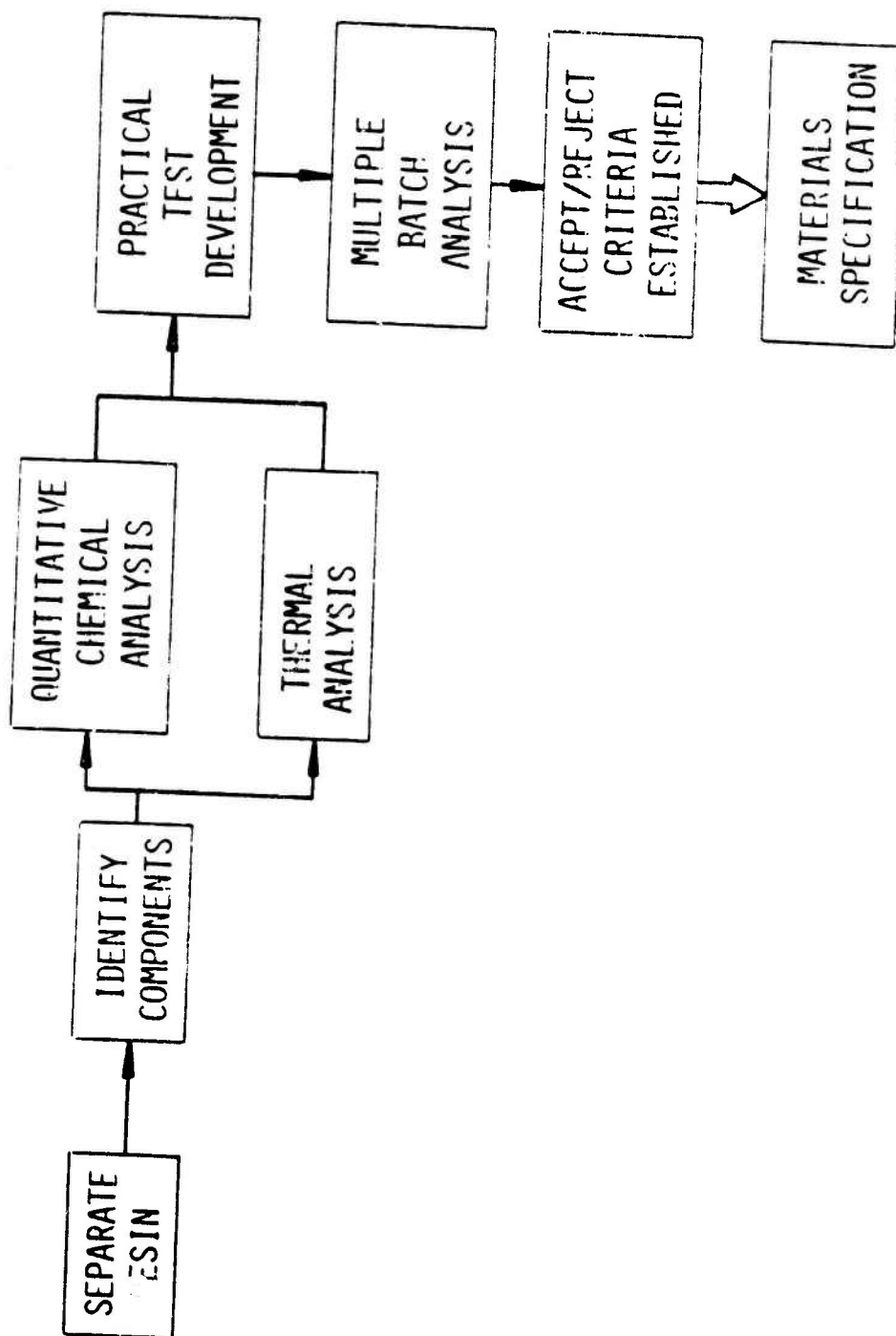


FIGURE 114. INSTRUMENTAL QUALITY CONTROL PLAN FOR PABST ADHESIVE AND PRIMER

STRUCTURAL ADHESIVE

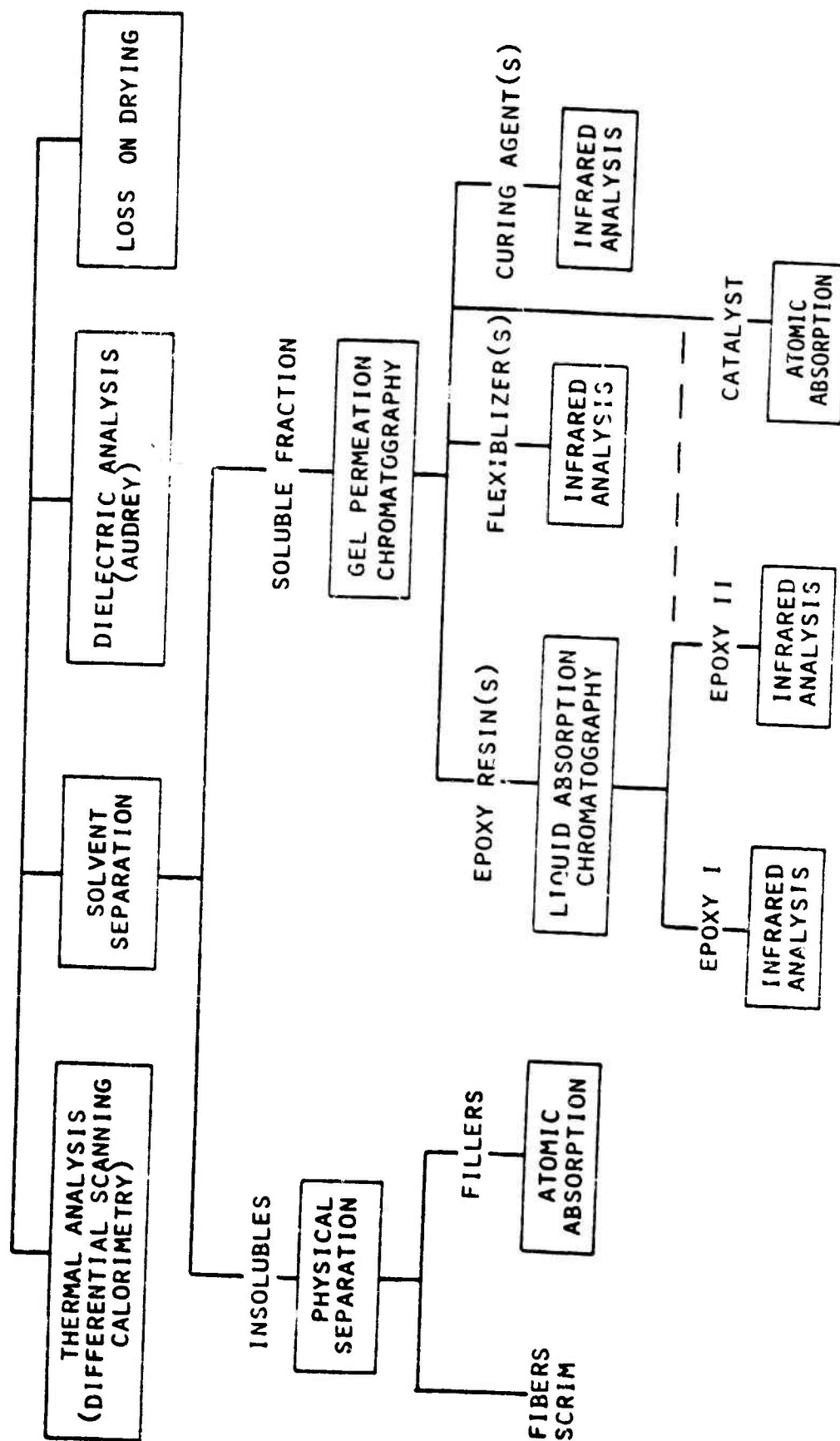


FIGURE 115. SEPARATION, IDENTIFICATION AND CHARACTERIZATION OF PABST ADHESIVE AND PRIMER SYSTEMS

SECTION VIII

AUXILIARY TEST PROGRAMS

8. Introduction

The auxiliary tests are continuing portions of other tasks performed in the program, and additional tasks added as part of the normal update due to new information deemed necessary from previous test results. They are handled separately because they are investigations within themselves aimed at obtaining a specific bit of information.

Most of the auxiliary tests were performed using the selected FM/73/BR127 adhesive system and the phosphoric acid anodize. Some of the tests used other conditions and these will be noted.

8.1 Alclad Versus Nonclad Alloys

The original premise of the program was to use nonclad alloys in conformance with MIL-A-83377. Later in the program, it was desired to see what the effects of clad details would be when similar tests were performed on them. Two types of tests were performed, lap shear and wedge crack. The alloy for the lap shear specimens was alclad 7075-T6. The specimens were tested at ambient temperature and at ambient temperature after 30 day salt spray. The wedge crack specimens were fabricated from alclad and nonclad 7075-T6 and 2024-T3.

The lap shear specimens showed no degradation during the 30 days salt spray. The failure mode was cohesive.

The 7075-T6 clad wedge crack specimens exposed to salt spray had an adhesive mode of failure while the clad 2024-T3 had a cohesive mode of failure. Beach exposure tests, which are covered in the next section, verify these results.

The indication of these tests is that the cladding on 7075-T6 alloys may contribute to a lack of environment resistance of the bonded joint and that durability may not be entirely dependent on providing a superior surface treatment.

8.2 Beach Exposure Tests

Long term outdoor exposure tests are being performed for this program. These tests comprise a variety of specimen types, alloys, and adhesives. The results to date are given here.

The tests are performed at the Douglas Aircraft Company's El Segundo Sea Atmosphere Test Station located on the beach in El Segundo, CA. The site is approximately 50 yards from the Pacific Ocean and located within the boundaries of an oil refinery. It also lies below the take-off pattern of Los Angeles International Airport and is adjacent to a sewage disposal plant. Rainfall is light and concentrated in January and February with essentially none from May to November. Total rainfall averages approximately 12 inches per annum. Corrosive agents deposited on test specimens remain for long periods and are not subject to frequent removal by rainfall.

The test specimen types in this program consist of alclad and nonclad 7075-T6 tensile lap shear specimens and alclad and nonclad 7075-T6 and 2024-T3 wedge crack specimens. The lap shear specimens were exposed in a stressed and nonstressed condition. In addition, on one half of the specimens, the cut edges are exposed and the adhesive flashing has been removed to expose the adhesive glue line. The stressed specimens are loaded to 1500 psi or approximately 30% of the ultimate specimen test strength.

After one year exposure, the stressed and nonstressed lap shear specimens were removed from the test site for a determination of strength retention. Two lap shear specimens from each of the four exposure groups were tested for strength retention. Figure 116 indicates that the loss in strength due to exposure was minor. All specimens failed cohesively with minor edge corrosion on the 7075-T6 with exposed glue lines. The remainder of the specimens were replaced on the beach after checking the stress level exerted by the test fixtures. After the second year of exposure, very little additional effect was noted.

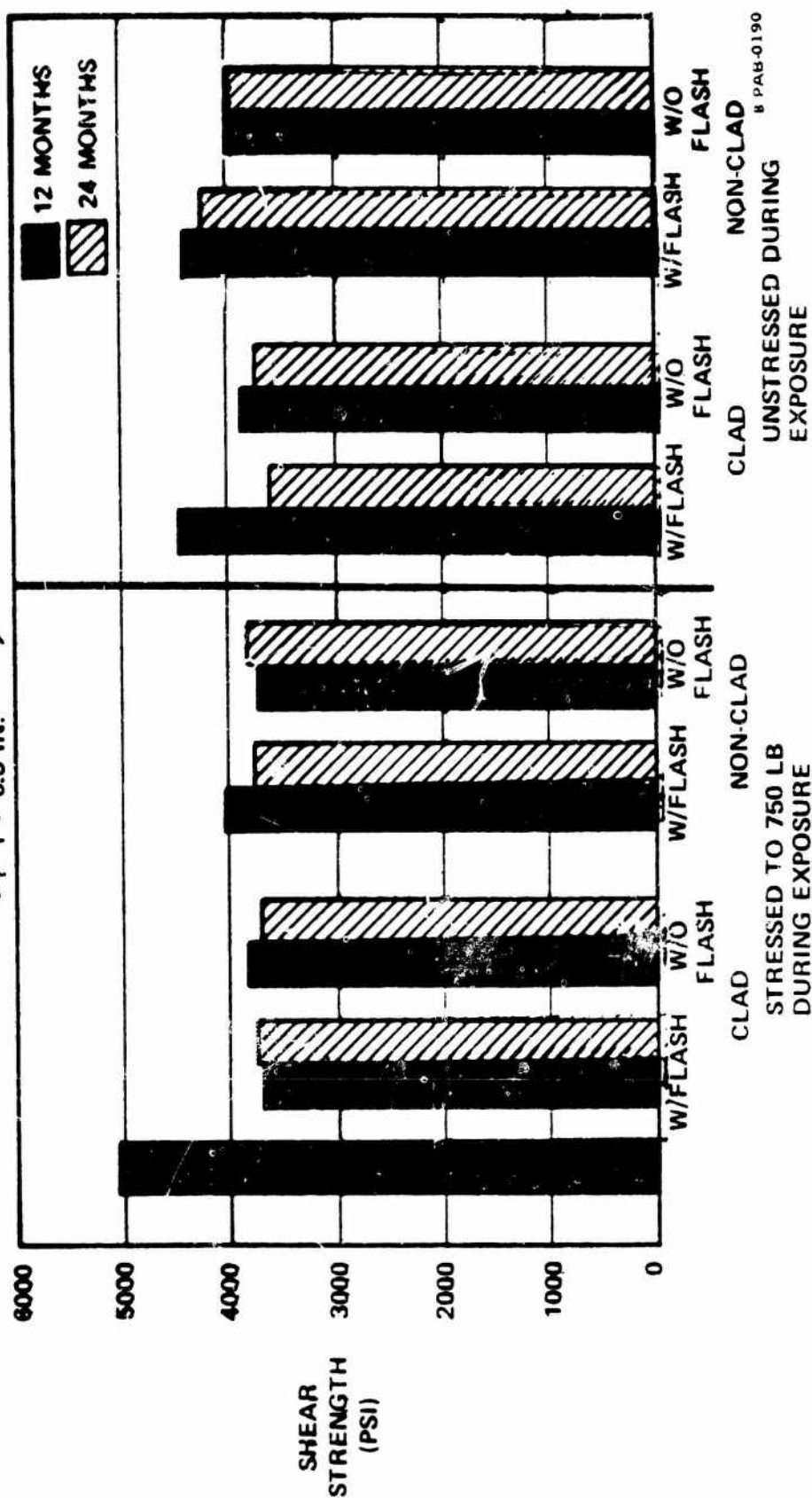


FIGURE 116. BEACH EXPOSURE TEST

Several groups of wedge crack specimens have been exposed at the beach site. Figure 117 graphs the crack growth on the alclad and nonclad 7075-T6 with two surface treatments, the phosphoric acid anodize and the optimized chromic acid anodize. The failures on the alclad 7075-T6 were very rapid and were an adhesive mode of failure. The nonclad specimens have indicated very little crack growth after 22 months at the beach site. The mode of failure has remained cohesive. It should be noted that no coatings other than the adhesive primer are on the specimens. The 2024-T3 alclad specimens were surface treated with two surface treatments, the optimized FPL etch and phosphoric acid anodize. The total time of exposure to date is 16 months. Both sets of specimens show negligible crack growth with the phosphoric acid anodize and the FPL etch. The specimens are still being exposed. Figure 117 shows the results to date. Fokker submitted some specimens fabricated using adhesives and processes used on the F28 aircraft. Their results are in Figure 118.

Fokker also processed some 7075-T6 alclad specimens using their chromic acid anodize which has no seal after anodizing. The specimens were bonded with FM73/BR127 and the Redux 775/BSL101 systems. After one month beach exposure, all specimens exhibited crack growths in excess of 1.5 inches. They were removed from the beach. All specimens exhibited an adhesive mode of failure. (See Figure 117.)

WEDGE CRACK SPECIMEN FM73 ADHESIVE FILM/BR127 PRIMER

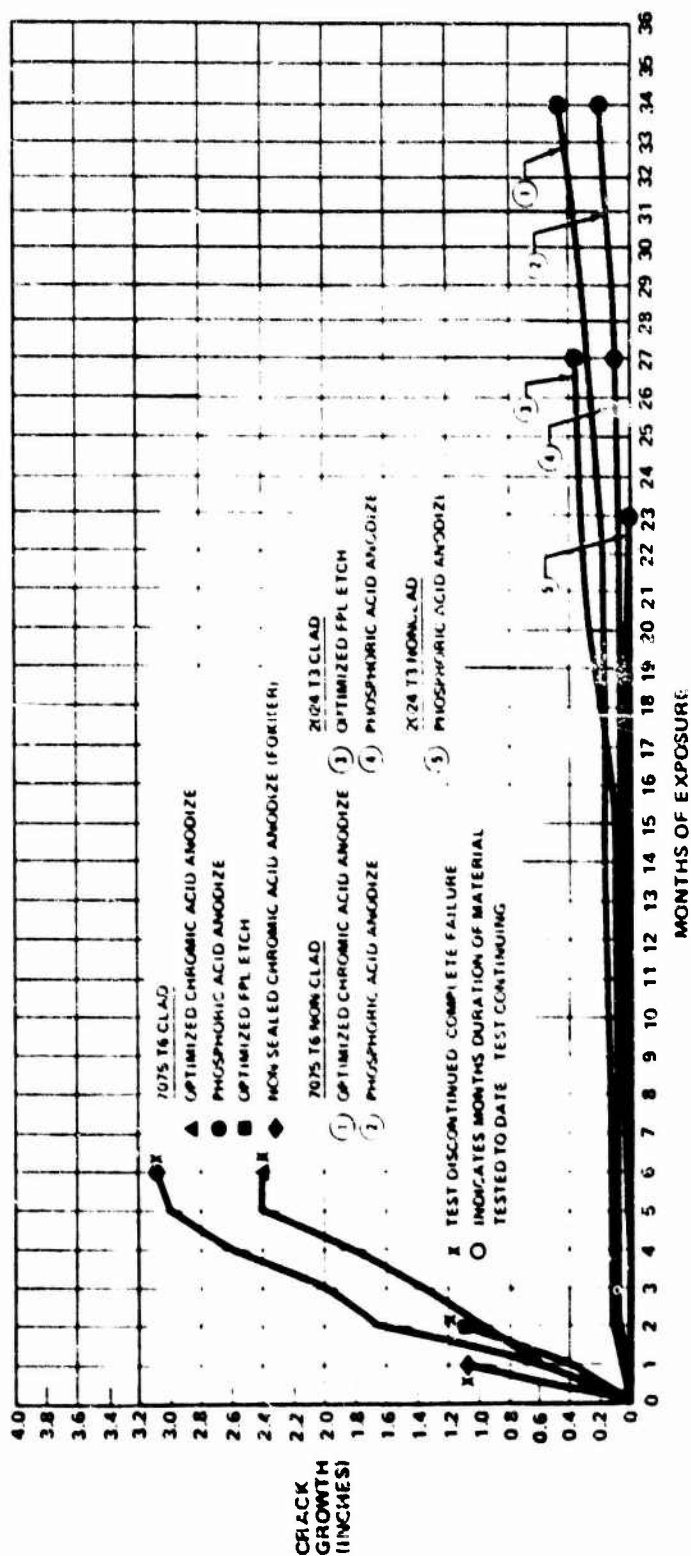
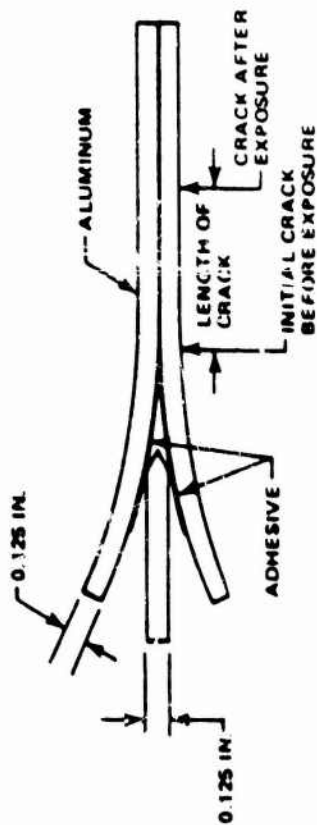


FIGURE 117. BEACH EXPOSURE TEST

OUTDOOR BEACH EXPOSURE

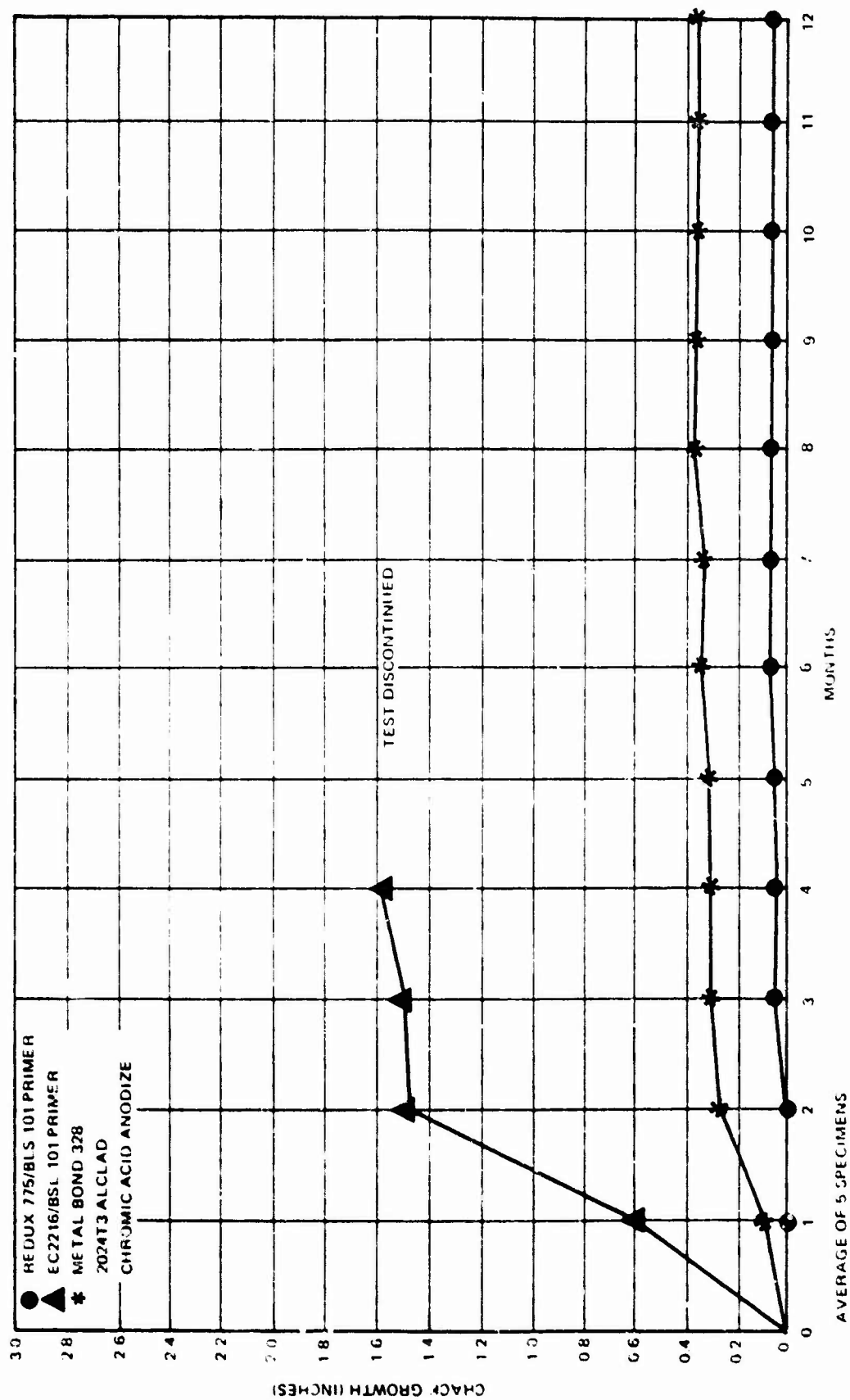


FIGURE 118. FOKKER VFW COORDINATION

8.3 Peel Ply Tests

At the beginning of the program, it was thought possible that some of the large panels may be of such a configuration that two bond cycles would be required to complete them. This type of operation would entail protecting the secondary bonding areas from excessive contamination during the first cure cycle and the preparation of those surfaces for the secondary bond cycle in a manner that would produce bonds equal in strength and quality to a single stage bond. Two methods were proposed. One was to leave the primed details in an unprotected manner and perform a solvent wipe cleaning after the first stage. It was felt that this type of function would be a hand operation with a commercial industrial solvent and a clean rag. Since these three items are relatively uncontrollable in a production atmosphere and since representative test specimens to verify the operation would be difficult to produce because of the variables involved, it was felt an alternate method should be investigated. One alternate method would be to cure a layer of adhesives in the secondary bond areas with some type of peel ply covering it.

The assembly could then be processed through the first stage and when the assembly was ready for the second stage bond, the peel ply could be removed exposing a fresh clean surface, more adhesive could then be applied and the second stage bond performed.

Peel ply materials investigated were aluminum foil, tight weave glass fabric and a Tedlar type release film.

Wedge crack specimens were fabricated per the above peel ply procedures. All tests showed a lack of adhesion between the two adhesive films after the secondary bond, except for the aluminum foil which was extremely difficult to remove from the first stage bond and considered impractical for production use.

Each of the adhesive suppliers were approached with the problem but to this date, no satisfactory material or method is available. All panels being bonded for the final article are one stage bonds.

8.4 Multiple Cure Cycles

The effect of multiple cure cycles on the properties of a bond joint was tested. The test was performed for the reason, as in Section 8.1.3., that more than one cure cycle may be required to meet the final configuration of the assembly.

The tests included lap shear specimens tested at ambient and 180°F after one and five cures and wedge crack specimens tested after each additional cure cycle through five cure cycles. A small reduction in shear strength was noted on the shear specimens tested at 180°F after exposure to five cure cycles. The wedge crack specimens did not indicate any effect after being exposed to each of the five cure cycles and all modes of failure were cohesive. Tables 40 and 41 give the results.

TABLE 40
MULTIPLE CURE CYCLE TEST

	TESTED AT AMBIENT TEMP.			TESTED AT 180°F		
	Specimen Ident.	Load (psi)	Failure Mode	Specimen Ident.	Load (psi)	Failure Mode
One Cure Cycle	224-1-1	4660	Cohesive with a slightly porous glue line	224-1-6	4106	Cohesive ↓
	-2	4877		-7	3816	
	-3	5490		-8	4197	
	-4	5447		-9	3695	
	-5	<u>5330</u>		-10	<u>4042</u>	
		5160 avg.			3971 avg.	
Five Cure Cycles	224-5-1	5433	Cohesive ↓	224-5-6	3350	Cohesive ↓
	-2	5258		-7	3253	
	-3	5120		-8	3406	
	-4	5185		-9	3330	
	-5	<u>4862</u>		-10	<u>3093</u>	
		5171 avg.			3286 avg.	

Tensile Lap Shear

Adhesive System: FM73 Adh. Film/BR127 Primer

Adhesive Wt.: .045 lbs/ft²

Surface Prep.: Phosphoric Acid Anodize

TABLE 41
MULTIPLE CURE CYCLE TESTS

	Specimen Ident.	Initial Crack Length	Total Crack Growth	Failure Mode
Number of Cure Cycles	224-1-1	2.18	2.30	Cohesive ↓
	-2	2.20	2.20	
	one -3	2.14	2.19	
	cure -4	2.20	2.25	
	cycle-5	2.23	2.28	
	224-2-1	2.17	2.25	Cohesive ↓
	-2	2.17	2.20	
	two -3	2.15	2.23	
	cure -4	2.13	2.21	
	cycles-5	2.14	2.20	
	224-3-1	2.21	2.25	Cohesive ↓
	-2	2.09	2.17	
	three -3	2.16	2.20	
	cure -4	2.11	2.17	
	cycles-5	2.15	2.23	
	224-4-1	2.16	2.22	Cohesive ↓
	-2	2.07	2.16	
	four -3	2.13	2.20	
	cure -4	2.11	2.16	
	cycles-5	2.16	2.23	
	224-5-1	2.20	2.26	Cohesive ↓
	-2	2.11	2.15	
	five -3	2.18	2.20	
	cure -4	2.12	2.18	
	cycles-5	2.13	2.17	

Wedge Crack at 140°F at 90-100% R.H. 1 Hour

Adhesive System: FM73 Adh. Film/BR127 Primer

Adhesive Wt.: .045 lbs/ft²

Surface Prep.: Phosphoric Acid Anodize

8.5 Anodizing on Chem-Milled Surfaces

During the program it was expected that some of the designs would include the anodizing and bonding to aluminum surfaces that had been processed by chem milling. Since we had no knowledge on the performance of phosphoric acid anodize on surfaces that had been modified by the action of the chem mill solution, it was determined to be one parameter that should be investigated. Both 2024-T3 and 7075-T6 nonclad alloys were tested. The types of test specimens were lap shear tested at ambient temperature, and wedge crack specimens tested at 140°F and 95-100% RH. The specimens were chem milled as in the normal production chem mill facility and then processed through the phosphoric acid anodize like a normal nonchem milled skin. The specimens were then primed and bonded with FM73/BR127 adhesive system. No effect was observable from this type of processing. Test results were comparable to specimens tested on nonchem milled skins. Tables 42 and 43 give the results of these tests.

TABLE 42
CHEM MILL SURFACE EFFECTS

	Specimen Ident.	Initial Crack Length	Total Crack Growth	Failure Mode
7075T6 Bare Alum. Alloy	225-1	.221	2.25	Cohesive ↓
	-2	2.18	2.23	
	-3	2.21	2.48	
	-4	2.22	2.25	
	-5	2.20	2.26	
2024T3 Bare Alum. Alloy	225-6	2.24	2.27	
	-7	2.17	2.22	
	-8	2.07	2.10	
	-9	2.11	2.17	
	-10	2.15	2.16	

Wedge Crack at 140°F at 95-100% R.H. 1 Hour

Adhesive System: FM73 Adh. Film/BR127 Primer

Adhesive Wt.: .045 lbs/ft²

Surface Prep.: Phosphoric Acid Anodize

TABLE 43
CHEM MILL SURFACE EFFECTS

	Specimen Ident.	Loads (psi)	Failure Mode
7075-T6 Bare Alum. Alloy	225-1	5729	Cohesive ↓ Y
	-2	5299	
	-3	5528	
	-4	5462	
	-5	5249	
		5453 avg.	
2024-T3 Bare Alum. Alloy	225-6	4879	Cohesive ↓ Y
	-7	4934	
	-8	5146	
	-9	5166	
	-10	5271	
		5079 avg.	

Tensile Lap Shear

Adhesive System: FM73 Adh. Film/BR127 Primer

Adhesive Wt.: .045 lbs/ft²

Surface Prep.: Phosphoric Acid Anodize

8.6 Bond Line Thickness Effects

On bonded assemblies, it can be expected that a variation in bond line thickness will exist. This test was intended to determine the effect of thickness variations and especially the mechanical properties of thicker than normal bond lines.

The types of tests performed were lap shear, T-peel and wedge crack.

The reduction in lap shear strength from 0.003 inch nominal thickness to 0.020 inch nominal thickness was 15% at ambient test temperature and 12% at 180°F test temperature.

The wedge crack specimens did not indicate any condition that would be unacceptable and all modes of failure were cohesive.

The T-peel specimens produced progressively higher peel strengths as the bond line thickness increased. The wedge crack, lap shear and T-peel results are expressed in Tables 44, 45 and 46.

TABLE 44
BOND LINE THICKNESS EFFECTS

Specimen Ident.	Bondline Thickness	Initial Crack Length Inches	Additional Crack After One Hour Exposure Inches	Failure Mode
226-1 -2 -3 -4 -5	.003" Nominal	2.21 2.21 2.16 2.03 2.09	.050 .080 .032 .034 .040	Cohesive ↓
226-6 -7 -8 -9 -10	.010" Nominal	2.02 1.99 2.13 2.00 1.97	No Exten .017 .037 .050 .008	Cohesive ↓
226-11 -12 -13 -14 -15	.020" Nominal	1.92 1.84 2.17 2.02 1.99	.065 .021 No Exten .025 No Exten	Cohesive ↓

NOTE: Test Environment one hour at 140°F and 95-100% R.H.

Wedge Crack Test

Adhesive system: FI173 Adh. Film/BR127 Primer
Surface Prep.: Phosphoric Acid Anodize
Metal Adherend: 7075-T6 Nonclad Aluminum

TABLE 4-5
BOND LINE THICKNESS EFFECTS

Specimen Ident.	Bond Line Thickness	Shear Strength psi	Failure Mode
Ambient Temp.	.003" Nominal	5964	Cohesive
		5803	
		5692	
		5662	
		5690	
		5762 avg.	
	.010" Nominal	5737	Cohesive
		5706	
		5829	
		5947	
		6017	
		5847 avg.	
	.026" Nominal	4747	Cohesive
		4984	
		4945	
		4868	
		4962	
		4901 avg.	
180°F	.003" Nominal	3623	Cohesive
		3986	
		4330	
		3751	
		3900	
		3918 avg.	
	.010" Nominal	3847	Cohesive
		4188	
		3801	
		3715	
		3859	
		3887 avg.	
	.026" Nominal	3611	Cohesive
		3397	
		3514	
		3328	
		3341	
		3438 avg.	

Tensile Lap Shear Test

Adhesive System: FM73 Adh. Film/PR127 Primer

Surface Prep.: Phosphoric Acid Anodize

Metal Adherend: 7075-T6 Bare Aluminum

TABLE 46
BOND LINE THICKNESS EFFECTS

Specimen Ident.	Bondline Thickness	T-Peel Strength In lbs/in.	Failure Mode
226-1-1 -2 -3 -4 -5	.003" Nominal	22 20 23 24 24 <u>22.5</u> avg.	Cohesive ↓
226-2-1 -2 -3 -4 -5	.010" Nominal	34 36 36 33 28 <u>33.5</u> avg.	Cohesive ↓
226-3-1 -2 -3 -4 -5	.020" Nominal	43 42 43 44 43 <u>43</u> avg	Cohesive ↓

AMBIENT TEMP.

Metal to Metal T-Peel

Adhesive System: FM73 Adh. Film/BR127 Primer

Surface Prep.: Phosphoric Acid Anodize

Metal Adherends: 2024-T3 Bare Aluminum

8.7 Effects of Defects Program

Normal production of large bonded assemblies can be expected to produce some level of nonuniform bond joints. These variations or nonuniformities can be caused by fit of details causing voids or porosity, inadvertent handling of details prior to primer application, variations in cure cycle, foreign objects in the bond line, and fractures caused by mishandling of the bonded assembly. The type of specimen used in this test program is a slow cycle fatigue specimen configured similar to the joints expected in the designed final article. The tests are still in progress and the results will be compiled at a later date. The program is outlined in Tables 47 and 48.

TABLE 47
EFFECT OF DEFECTS





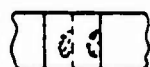
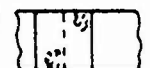

TEST NO	DEFECT SOURCE	DEFECT LOCATION AND ORIENTATION	NUMBER OF SPECIMENS
1	OVERAGED ADHESIVE	PLACE ADHESIVE IN CLEAN ROOM AT R.T. AND LOCAL HUMIDITY PERIODIC LAP SHEAR TESTS TO 80 PERCENT ALLOWABLE. USE THIS ADHESIVE FOR SPEC. FULL WIDTH	1
2	POROSITY (ii) HIGH HEAT RATE (iii) WET ADHESIVE CLEAN ROOM AT R.T. AND 100 PERCENT R.H. (iii) THICK GLUE LINE	PREPARE SPECIMENS WITH ADHESIVE IN THE CONFIGURATIONS AS LISTED IN LEFT HAND COLUMN. FLAW FULL WIDTH OF SPEC. NOTE: MEASURE STIFFNESS, I.E., LOAD VERSUS STRAIN FOR EACH CASE.	3
3	WHITE GLOVE	(i) FULL WIDTH CONTAMINATION (ii) THUMBPRINT APPROXIMATELY CENTERLINE OF SPECIMEN	2
4	BARE HAND	SAME AS FOR "WHITE GLOVE" ABOVE EXCEPT CONTAMINATION BY BARE HAND.	2
5	LOW BONDING TEMPERATURE	PREPARE SPECIMEN WITHOUT FLAWS AND THEN CURE AT 25°F LESS THAN OPTIMUM.	1
6	TOO LITTLE TIME TO CURE TEMPERATURE	THIS REPRESENTS A HIGH MASS POINT ARE WHERE CURE TEMPERATURE TAKES LONGER TO BE ACHIEVED. CURE TIME = 30 MIN	1
7	VARIABLE BONDLINE THICKNESS	(i)  VARIABILITY AS SHOWN ON BOTH SIDES (ii)  UNIFORM AS SHOWN OVER FULL WIDTH OF SPECIMEN	1
8	FOREIGN OBJECTS	AREAS OF POROSITY AROUND WIRES 	2

TABLE 48
EFFECT OF DEFECTS

TEST NO.	DEFECT SOURCE	DEFECT LOCATION AND ORIENTATION	NUMBER OF SPECIMENS
9	VOIDS	<p>(i)  FLAW 50% BOND AREA, ONE SIDE.</p> <p>(ii)  1-IN. DIA FLAW, ONE AT EDGE AND ONE AT CENTERLINE, ONE SIDE ONLY.</p> <p>(iii)  1-IN. DIA FLAW, AT EACH EDGE, ONE SIDE ONLY.</p>	3
10	FRACTURED BONDLINE	 BONDLINE FRACTURE FRACTURE BONDLINE 1/4 IN. WIDE	1

8.3 Thick Adherend Fatigue Test

At the very beginning of the program a test was performed which was called a "show stopper." This test was to determine if a bond joint exposed for a reasonable period of time to a hostile environment and then exposed to a fatigue test would fail in a period of time that would preclude proceeding with the rest of the program.

A group of thick adherend lap shear specimens were fabricated using the phosphoric acid anodize, FM73 and BR127 adhesives. The adherends were fabricated from .500 inch nonclad 7075-T6 plate. After fabrication, the specimens were exposed to 140°F and 95-100% relative humidity for 69 days.

8.8 (Cont'd)

The tests performed were:

- a. Static test at room temperature to determine ultimate strengths.
- b. Cyclic fatigue test at 1800 cycles per minute (CPM) at a load of 30% ultimate and a low cycle load of $R = .2 = 6\%$ ultimate.
- c. Cyclic fatigue test at 1800 CPM at 30% ultimate and a compression alternate of $R = -.2$
- d. Cyclic fatigue as in b. except the test temperature was -50°F .

The ultimate static shear strength was 5110 psi average. All the fatigue specimens tested exceeded 10 million cycles without failure. The results are in Table 49.

8.9 Wedge Crack Environmental Test

Additions to the environment resistance test program were made after the original test plan had been submitted. These additions were made after contact with the Air Logistics Command and visits by a group from the PABST team.

The tests were to determine the resistance of the bond joint to exposure to common materials used at field and depot level facilities. These were an alkaline cleaner (MIL-C-25769) used for washing the aircraft, and a representative bilge fluid. The effort to obtain a representative bilge fluid was not accomplished until the PABST group trip to Europe in November 1975. The selection of a representative bilge fluid was taken from "The Corrosion Properties of Airframe Contaminants" published by the Ministry of Defense, United Kingdom, December 1971. The bilge fluid selection was based on the results of tests in which an aluminum sample was placed in the test solution of a period of one week under vibration of 70 CPS vertical oscillation. Aluminum sample weight loss was then calculated. The fluid selected for the bilge fluid test showed the greatest weight loss in the test specimen after this time period. The test solution consists of distilled water, 0.46% sodium sulfate, 0.82% sodium chloride, and sodium hydroxide to adjust solution to a pH of 7.8.

TABLE 49
THICK ADHEREND FATIGUE TESTS

Static at room temperature

Specimen Number	Loads	PSI	Failure Mode
7P2B - 2 7P2B - 3	2480 2655	4909 5312 5110 avg.	Cohesive Cohesive

Tension/Tension at room temperature

30% ULT/R = + .2

Specimen Number	Number of Cycles	Failure Mode
7P2B - 4 7P2B - 5	12,752,000 10,165,000	No Failure No Failure

Tension/Compression at room temperature

30% ULT/R = - .2

Specimen Number	Number of Cycles	Failure Mode
7P2C - 1 7P2C - 2	10,180,000 10,300,000	No Failure No Failure

Tension/Tension at -50°F

30% ULT/R = + .2

Specimen Number	Number of Cycles	Failure Mode
7P2C - 3 7P2C - 4	10,000,000 Test Discontinued	No Failure -

Exposure = 69 days at 140°F and
95-100% R.H.
7075-T6 nonclad
alloy

3.9 (Cont'd)

Wedge crack specimens were placed in a container with the liquid/air interface maintained in the wedged test area of the specimen. Equipment to vibrate the specimens as specified above was not available; instead, the solution was constantly agitated to create some movement.

At the completion of the test, all specimens were measured for any increase in crack length and then separated to examine the failure mode. See Table 50 for results of the wedge crack tests. All specimens exhibited a 100% cohesive failure with no apparent solution attach on the exposed bond line. Indications of corrosion was apparent along the exposed cut edges of the specimens that had no surface protection (phosphoric acid anodize or primer) at the liquid/air interface. This corrosion condition was similar to that experienced in the tests referenced by the Ministry of Defense in their document.

No effect was noticed from 7 days immersion in the MIL-C-25769 alkaline cleaner. All modes of failure in the bond joint were cohesive. See Table 51 for test results.

TABLE 50
HEDGE CRACK ENVIRONMENTAL TEST

Adhesive System	Specimen Ident.	Initial Crack Length, Inches	Total Crack Length, Inches	Failure Mode
FM73 / DR127	230-1-31 -32 -33 -34 -35	2.14	2.24	Corrosion on cut edge of specimen at liquid air interface and cohesive failure
		2.15	2.15	
		2.18	2.18	
		2.18	2.18	
		2.22	2.27	
EA9628 / EA9202	230-2-31 -32 -33 -34 -35	2.03	2.12	
		2.10	2.19	
		2.14	2.22	
		2.12	2.24	
		2.14	2.20	
AF55 / EC3950	230-3-31 -32 -33 -34 -35	2.07	2.15	
		2.07	2.18	
		2.08	2.17	
		2.03	2.18	
		2.05	2.19	
H1133 / H6740	230-4-31 -32 -33 -34 -35	2.03	2.12	
		2.04	2.14	
		2.04	2.08	
		2.04	2.11	
		2.03	2.10	

Surface Preparation: Phosphoric Acid Anodize

Metal Adherends: 7075-T6 Bare

Environmental Exposure: 7 Days Immersion Bilge Fluid

at Ambient Temp 70 CPS Vibration

TABLE 51
WEDGE CRACK ENVIRONMENTAL TEST

Adhesive System	Specimen Ident.	Initial Crack Length, Inches	Total Crack Length, Inches	Failure Mode
FM73 / BK127	230-1-36 -37 -38 -39 -40	2.19	2.25	Cohesive
		2.21	2.21	
		2.16	2.16	
		2.60	2.60	
		2.18	2.18	
EA9628 / EA9202	230-2-36 -37 -38 -39 -40	2.13	2.25	
		2.20	2.24	
		2.17	2.29	
		2.17	2.29	
		2.19	2.25	
AF55 / EC3950	230-3-36 -37 -38 -39 -40	2.05	2.13	
		2.03	2.15	
		2.06	2.19	
		2.02	2.20	
		2.10	2.25	
M1133 / M674G	230-4-36 -37 -38 -39 -40	2.01	2.23	
		2.09	2.18	
		2.02	2.16	
		2.12	2.22	
		2.04	2.13	

Surface Preparation: Phosphoric Acid Anodize

Metal Adherends: 7075-T6 Bare

Environmental Exposure: 7 Days Immersion at Ambient Temp. in MIL-C-25709 Alkaline Cleaner

8.10 Acidified Salt Immersion (Immersion Corrosion)

This test was performed to determine the resistance of bonded specimens to a 5% acidified salt solution. The specimens were tensile lap shear fabricated from 7075-T6, -T73 and -T76 non clad alloys. T-peel specimens were fabricated from 2024-T3 nonclad alloy. This test was to determine if the heat treatment of the alloys would make a difference in the resistance of a bond joint to a hostile environment.

The specimens were treated with three types of surface treatment; optimized FPL etch, phosphoric acid anodize and the optimized chromic acid anodize. FM73 and BR127 were used in all of the tests. The immersion period was 30 days. See Table 32 for composition of the acidified salt solution.

The 2024-T3 shear specimens showed a definitely higher shear strength for the chromic anodize (5021 average) over the FPL etch (3681 average) and the phosphoric acid anodize (3904 average).

These differences are difficult to explain since all the modes of failure on the specimens were cohesive with no evidence of bond line corrosion. The results of the tests are in Tables 52 and 53.

TABLE 52
ACIDIFIED SALT IMMERSION
Lap Shear

Specimen Ident.	Surface Treatment	Aluminum Alloy	Tensile Shear Strength (psi)	Failure Mode
231-1 -2 -3 -4 -5	FPL	7075-T6	3757 3611 3658 3739 3643 <u>3681</u> avg.	Cohesive ↓
231-6 -7 -8 -9 -10	Phosphoric Acid Anodize	7075-T6	3800 3658 3638 3692 3914 <u>3740</u> avg.	Cohesive ↓
231-11 -12 -13 -14 -15		7075-T73	4198 3929 4049 4132 3908 <u>4043</u> avg.	Cohesive ↓
231-16 -17 -18 -19 -20		7075-T76	4156 3654 3847 4040 3942 <u>3929</u> avg.	Cohesive ↓
231-21 -22 -23 -24 -25		7075-T6	4852 4921 4838 4795 4853 <u>4851</u> avg.	Cohesive ↓
231-26 -27 -28 -29 -30	Chromic Acid Anodize	7075-T73	5025 4984 4969 5110 5150 <u>5047</u> avg.	Cohesive ↓
231-31 -32 -33 -34 -35		7075-T76	5191 4980 5470 5131 5055 <u>5165</u> avg.	Cohesive ↓

NOTE: All specimens exhibited porous glue lines. No signs of bond line corrosion.

Tensile Lap Shear

Adhesive System: F1173 Adh. Film/BR127 Primer

Environmental Exp.: 30 Day Immersion Acidified 5% Salt Solution

TABLE 53
ACIDIFIED SALT IMMERSION
T-Peel

Specimen Ident.	Surface Treatment	Peel Strength in Lbs./In.	Failure Mode
231-1 -2 -3 -4 -5	FPL	9.5 10.5 11.3 12.0 11.5 11.0 avg.	Cohesive
231-6 -7 -8 -9 -10	Phosphoric Acid Anodize	9.5 10.5 13.3 10.0 12.5 11.0 avg.	Cohesive
231-11 -12 -13 -14 -15	Chromic Acid Anodize	14.0 13.0 9.5 12.5 9.0 12.5 avg.	Cohesive

NOTE: All specimens exhibited porous glue lines. No signs of bond line corrosion.

Metal to Metal T-Peel

Adhesive System: FM73 Adhesive Film/BR127 Primer

Metal Adherent: 2024-T3 Bare

Environmental Exposure: 30 Days Immersion Acidified 5% Salt Solution at Ambient Temp.

8.11 Neat Adhesive Tests

The neat adhesive tests are a separate function of the program and are intended to characterize the adhesives. Paragraph 6.3.1.8 covers a portion of the program.

8.12 Bond Line Moisture Absorption

This test was intended to determine the moisture penetration rate of an adhesive in a simulated bond line. Precured samples of the adhesive were preweighed and clamped between aluminum adherends, with rubber sheets separating the adhesive and the adherend, to form a representative bond line. The specimens were then exposed to 140°F and 95-100% relative humidity. Each seven days, the specimens were removed from the environment and weighed. The adhesives used in this test were:

- FM73 with a mat carrier
- EA9C28 with a woven carrier
- AF55 with a woven carrier
- M1133 with a woven carrier
- M1133 with a mat carrier
- PL729 with a woven carrier
- FM47 with a woven carrier

The results, after ten weeks of exposure, are expressed in Figures 119 and 120. The FM73 and PL729 were the slowest adhesives to reach an equilibrium point in moisture absorption. Also, the percent increase in weight on these two adhesives was the least. FM47 had a reverse effect and lost weight during the exposure period. It is not known at this time the reason for this weight loss.

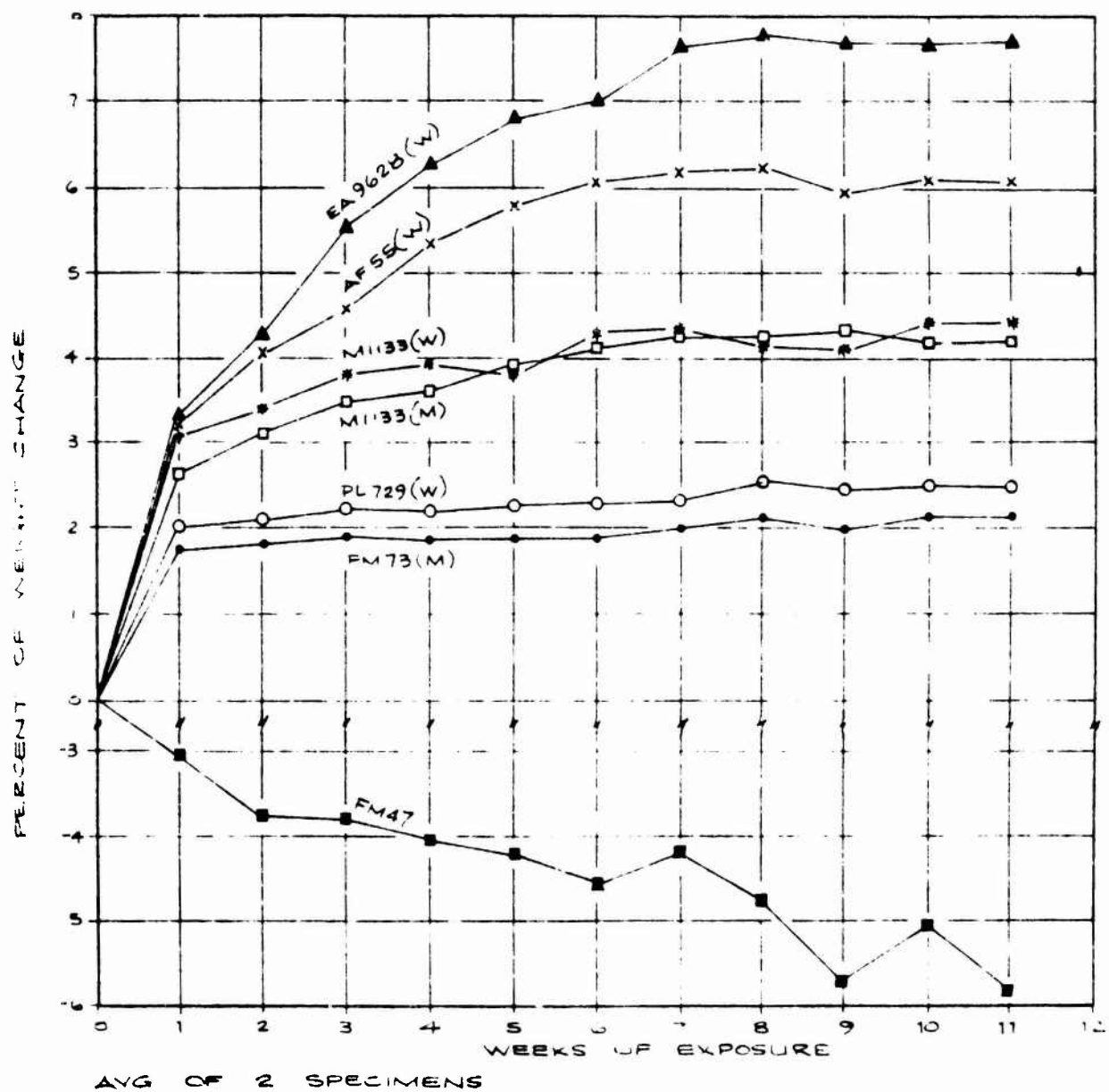


FIGURE 119. BOND LINE MOISTURE ABSORPTION - WEIGHT CHANGE

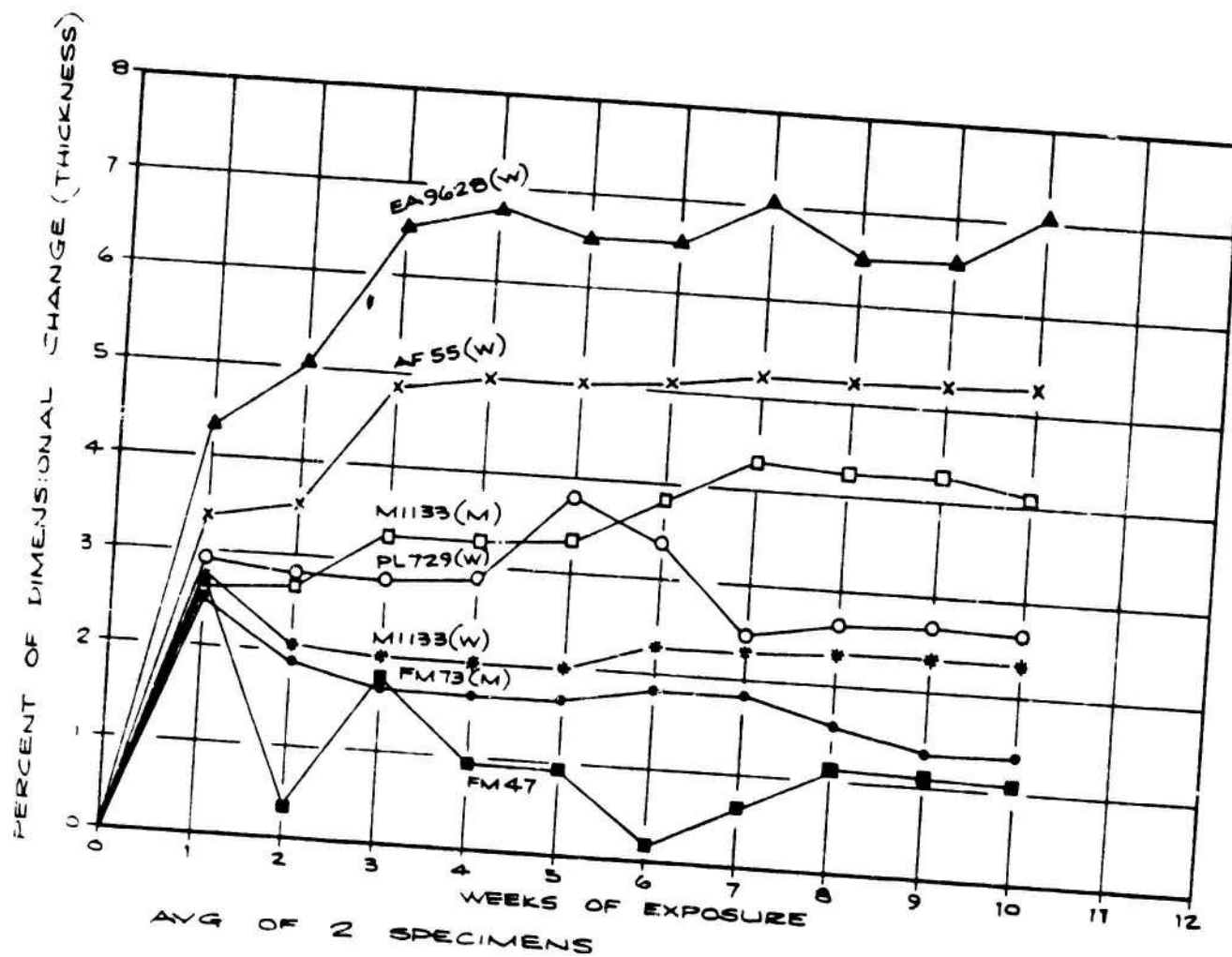


FIGURE 120. BOND LINE MOISTURE ABSORPTION - DIMENSIONAL CHANGE

8.13 BR127A Adhesive Primer Versus BR127

It was known that Lockheed Aircraft used a version of BR127 primer that was supposed to produce a higher peel strength when bonded with FM73, especially at the lower temperatures. Tests performed to compare the two systems were lap shear at room temperature, 180°F and -70°F, peel at -70°F and wedge crack specimens.

The BR127A had a higher peel strength than the BR127 at the thinner thicknesses but the same peel at the maximum allowed primer thickness. This may indicate that the peel strength of BR127A is much more sensitive to thickness variations than the BR127. The results are in Figures 107 and 109.

The wedge crack specimens all produced acceptable cohesive failure. Exposure to 30 day salt spray of BR127A coated specimens showed no penetration of the primer or surface corrosion. A 10 minute exposure to the chem mill solution did not penetrate the primer.

8.14 Fire Extinguisher Fluid Exposure

Wedge crack specimens fabricated with the four candidate adhesive systems have been immersed in a fire extinguisher test fluid for a period of seven days. Fire extinguisher test fluid consists of a 6% by volume solution of Aero-O-Foam liquid mechanical foam and water. This material is used to foam runways in preparation for emergency landings, in addition to being used for extinguishing fires. These types of materials can be very corrosive when they are in contact with the aircraft fuselage for long periods of time.

After seven days exposure, the wedge crack specimens on all the adhesive indicated a cohesive mode of failure with a maximum crack growth of .08 inch.

8.15 Cold Bond Adhesives

At one time in the program, the possibility existed that secondary splice doubler bonding may be part of the design. This type of bonding is being used by Fokker to join large bonded assemblies prior to installation of mechanical attachments.

8.15 (Cont'd)

The materials evaluated were two part epoxy room temperature curing adhesives. They were EA9309, EA9320, EA934 and EC2216. The first three are Hysol materials, the EA934 being 350°F resistant. EC2216 is manufactured by Minnesota Mining and Manufacturing Co. It is the material presently in use by Fokker.

The tests performed were lap shear at room temperature, +180°F and -70°F and at room temperature after immersion for 120 days in TT-S-735, JP-4, MIL-H-5606, and seven days in fire extinguisher fluid.

T-peel specimens were tested at room temperature and -70°F. Wedge crack specimens were tested at 140°F and 95-100% relative humidity and after immersion in the fluids used in the lap shear tests.

All the specimens were phosphoric acid anodized and primed with BR127.

The lap shear test results are in Figure 121. EA9320 and EA934 had the best shear strength at +180°F. All of the adhesives exhibited about the same resistance to the fluid immersions; the average shear strength of EC2216 was lower than the other materials. Figure 122 gives the T-peel strength at room temperature. All the materials exhibited a low shear strength at -70°F. Figure 123 shows the wedge crack growth of the various adhesives after immersion in the test fluids.

EA9320 showed some lack of resistance to 140°F and 95-100% relative humidity while EA934 had extensive crack growth with some failure between the adhesive and the primer. The three Hysol materials performed well in the fluid immersions but the EC2216 exhibited large crack extensions in all of the fluids.

For use on the PABST program the selection was narrowed to EA9309 and EA9320 when a design decision was made to delete the use of cold bond adhesives from the final article and further testing was not continued.

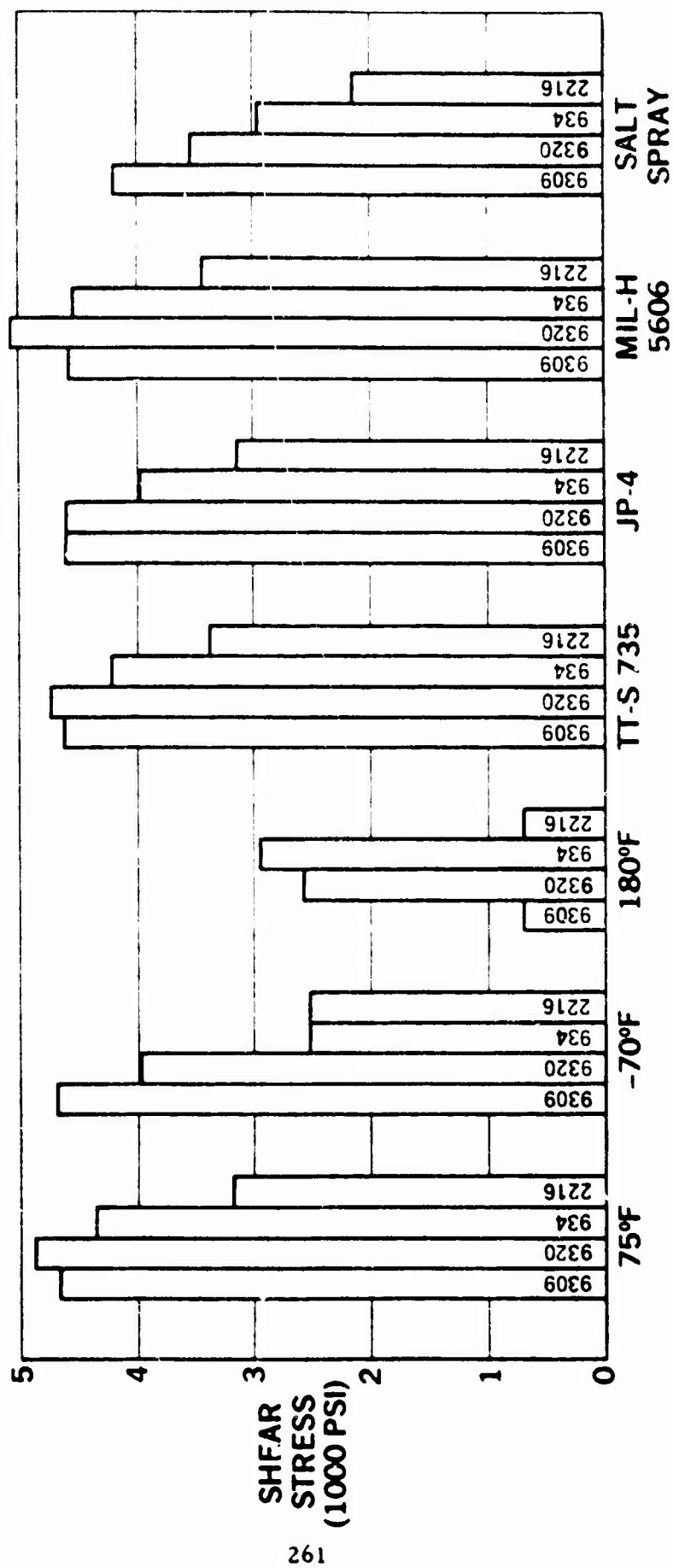


FIGURE 121 COLD BOND LAP SHEAR

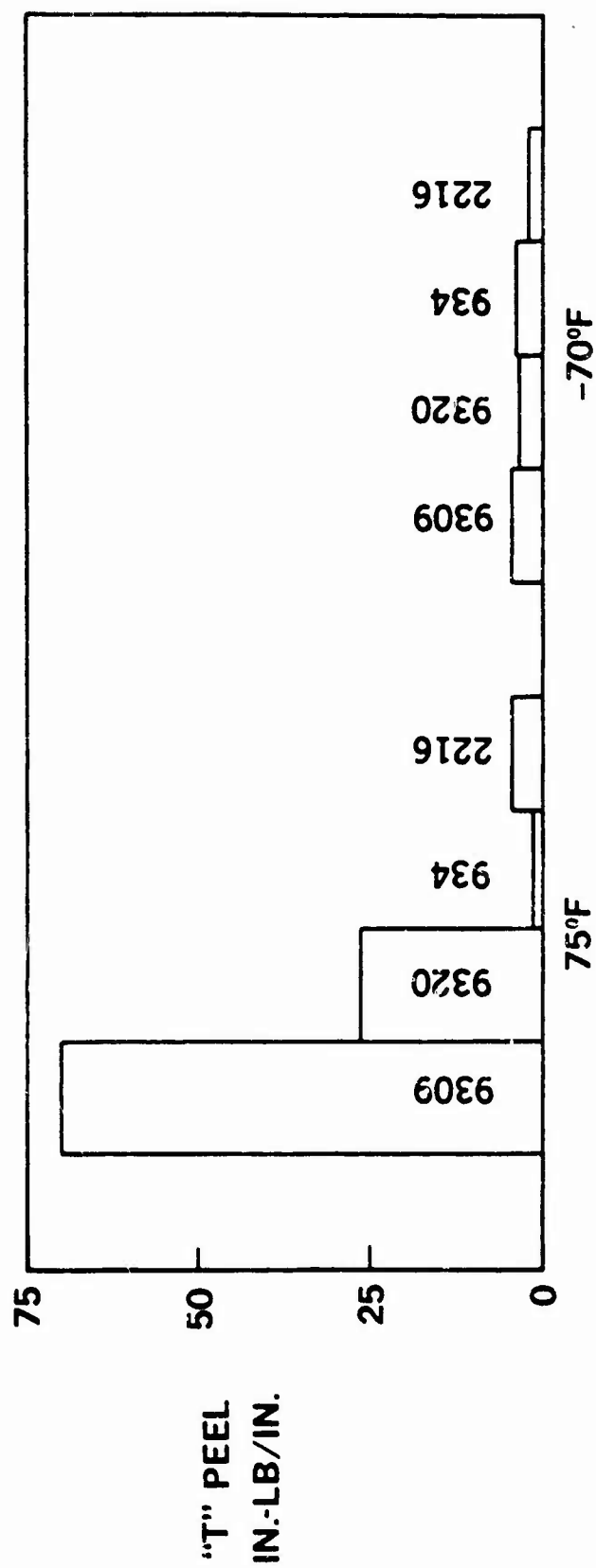


FIGURE 122. COLD BOND "T" PEEL

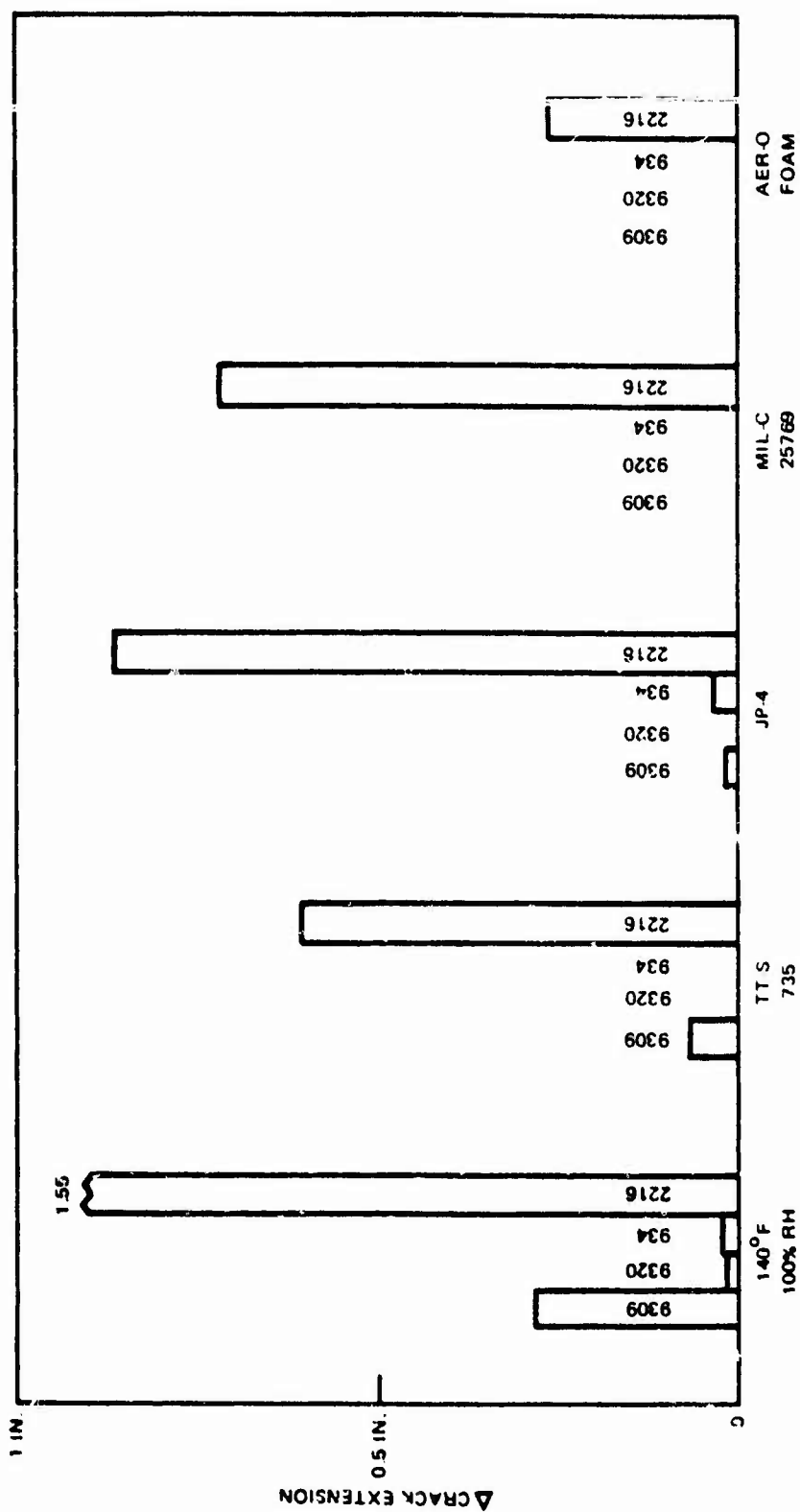


FIGURE 123. COLD BOND WEDGE CRACK

8.16 RAAB Tests on Adhesive with Alternate Carriers

The original test program on RAAB cyclic stressed specimens in a elevated temperature and moist environment indicated the adhesive system with the mat carrier had better survivability than those with woven carriers. This effect was also evident in the double cantilever beam tests. Samples of each adhesive were obtained with both types of carriers from the manufacturers. RAAB specimens were fabricated using the phosphoric acid anodize and both BR127 and EC3950 primer systems. Tests were conducted at 1500 psi and 900 psi in a test chamber maintaining 140°F and 95-100% relative humidity. The load cycle rate was 4 cycles per hour.

Figures 124 and 125 depict the results of the tests for each combination of adhesive, carrier and primer. The results show that at the lower stress levels, the woven carrier adhesives are more durable than the mat carrier adhesives. The BR127 primer was marginally better than the EC3950.

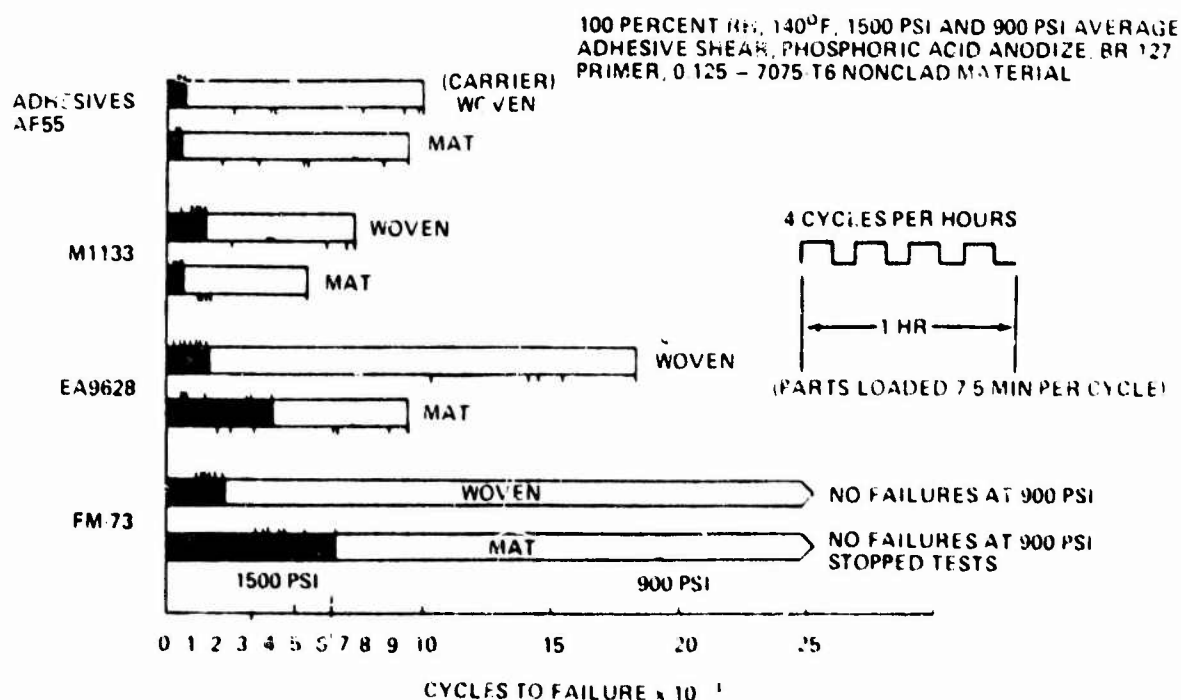


FIGURE 124. RAAB SPECIMENS ALTERNATE CARRIERS BR127 PRIMER

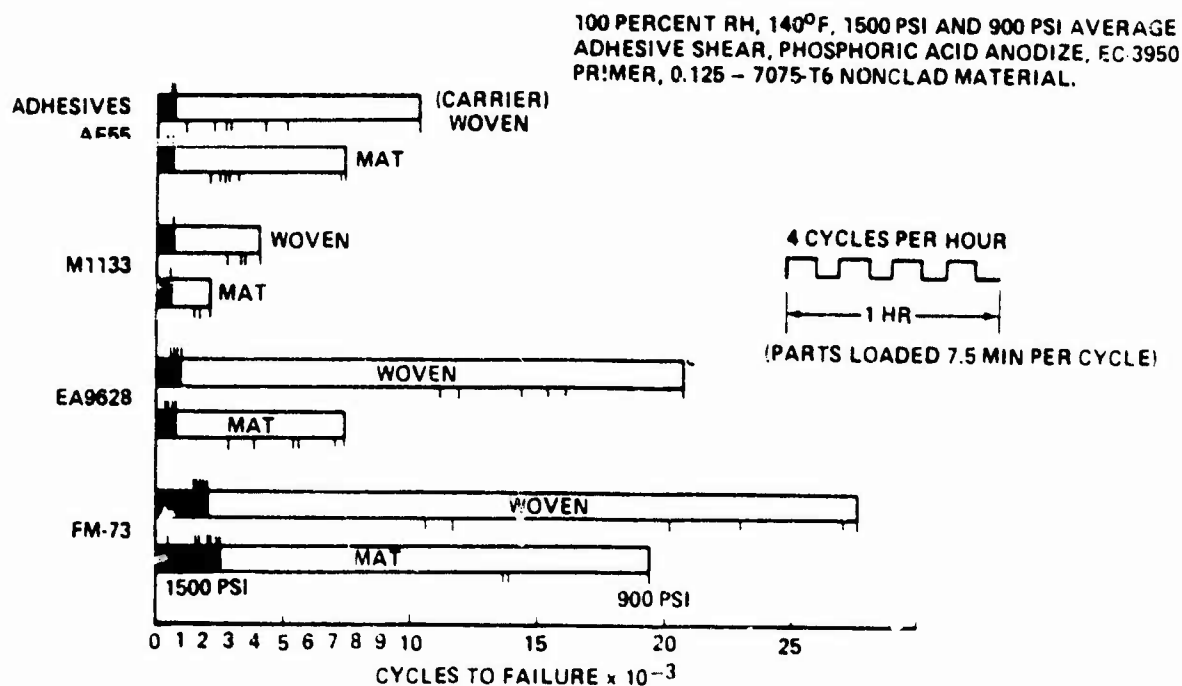


FIGURE 125. RAAB SPECIMENS ALTERNATE CARRIERS EC3950 PRIMER

Figure 126 depicts tests run by the Hysol Division of the Dexter Corporation, the manufacturers of EA9628. Several different carriers were used in these tests. The results indicate a closer similarity between the FM73 and the EA9628 adhesive but all the results on both adhesives are lower or less durable than the DAC results. Part of this difference could be attributed to a wider notch in the Hysol specimens. A second consideration that can be made is that the moisture content of the adhesive prior to bonding may produce the single most important variable in this type of test. Additional testing is being proposed to get a better understanding of this effect.

The Hysol data was obtained from a report LR-177-67 dated 4-25-77 written by Hysol Division, Dexter Corporation, Pittsburg, California.

100 percent RH, 140°F, 1500 psi and
 900 psi average adhesive shear phosphoric
 acid anodize, BR127 primer, 0.125 - 1075-T6
 nonclad metal

Adhesives

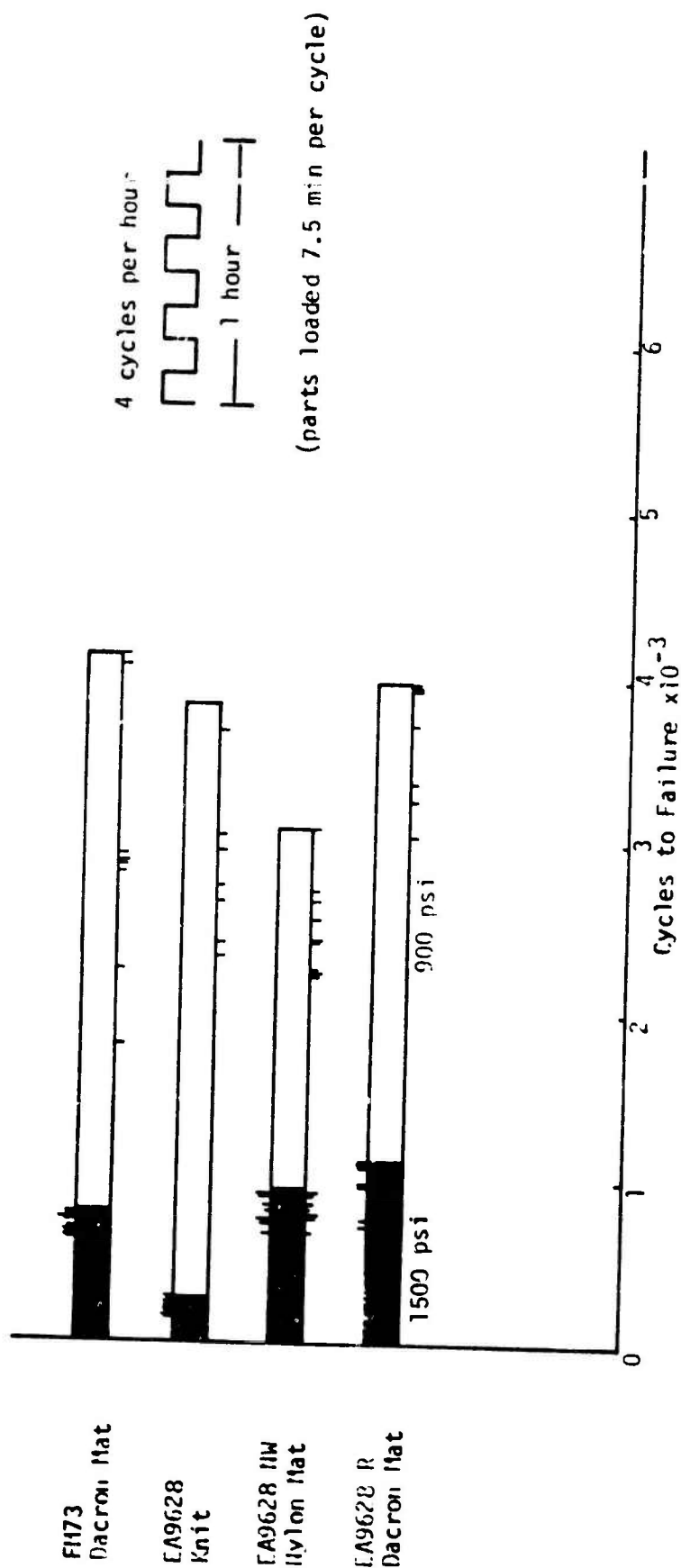


FIGURE 126. RAAB SPECIMENS HYSOL TESTS

8.17 Bond Line Thickness Control

The control of the bond line thickness is important at the edge of details since this is the highest stress point of the total bond area. In normal fabrication of assemblies, the adhesive flow allows the adherends to "pinch off" at the edge of details producing a very thin bond line. Structural Mechanics Engineering requested that tests be performed to regulate the bond line thickness in this area.

The methods first tried were the use of shims or dams around the edges of the adherends, a reduction in bonding pressure during cure, and the use of a controlled flow adhesive. The shim or dam method could control the pinch-off effect to some extent, however the number of shims required for each assembly and the proper location of them is not practical in production.

Reducing the bonding pressure would increase the level of voids, porosity, and create thick bond lines in other areas of the assembly. The controlled flow adhesive would have required a basic manufacturing change in the adhesive manufacture which would have required a complete retest.

Subsequent to the above program, samples of adhesive BSL308A from Ciba Geigy, FI173 from American Cyanamid and EA9328 from Hysol have been provided with glass beads in the adhesive. Tests indicate that these materials do maintain a relatively constant bond line thickness to the edge of the details.

APPENDIX "A"

Chemical Analysis and Solution Control

I. Alkaline Cleaner - Turco 4215S

Lab Tank Size

Tank Size - 9.75 Inches wide x 19 Inches long x 25.75 Inches deep

Solution Volume with 24.75 Inches deep = 19.84 gallons

Solution Makeup

Turco 4215S Cleaner - 11.31 pounds

Turco 4215 Additive - 200 ml

Deionized Water - Remainder

Operating Temperature - 145°-155°F

LABORATORY CONTROL PROCEDURE FOR TURCO 4215

1. Obtain a sample from the bath and allow to cool.
2. Pipette a 50 ml sample into a beaker and add approximately 50 ml distilled water.
3. Add 5 to 8 drops Brom Cresol Green Indicator.
4. Titrate with 1.0 N (normal) H_2SO_4 (sulfuric acid) until color changes from blue-green to yellow. If a pH meter is available, titrate to pH 4.0.
5. Record number of ml's of 1.0 N H_2SO_4 used.
6. Calculation:

$$(\text{No. of ml's } 1.0 \text{ N } H_2SO_4) \times 0.48 = \text{oz/gal Turco 4215}$$

7. Example:

15.3 ml of 1.0 N H_2SO_4 used.

$$15.3 \times 0.48 = 7.3 \text{ oz/gal Turco 4215.}$$

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ALKALINE CLEANER TANK RECORD

Date of Analysis	4215 Concentration oz/gal	Temp °F	Additions
2-20-75	9.12	140	
3-03-75	8.2	155	
3-24-75	7.4	100	200 ml Additive
4-04-75	7.1	-	100 ml Additive
4-17-75	7.54	145	
5-02-75	7.13	150	
5-12-75	6.77	148	200 ml Additive
5-21-75	6.79	151	62 grams Turco 4215
6-02-75	6.22	152	65 grams Turco 4215
6-10-75	6.96	150	
6-19-75	6.86	152	Partial dump - 700 grams 4215
6-20-75	-	-	500 ml Additive
6-30-75	6.14	-	400 grams 4215
7-08-75	6.72	123	100 grams 4215
7-14-75	7.22	151	

2. Deoxidizing Solutions

a. Bell Helicopters per MBFW108

Lab Tank Size

Tank Size - 9.25 Inches wide x 18.5 Inches long x 25.75 Inches deep

Solution Volume with 24.75 Inches deep = 18.34 gallons

Solution and Operating Condition

H_2SO_4 - 22 to 28% by weight

$Na_2Cr_2O_7$ - 2 to 3% by weight

Temperature - 140° to 160°F

Immersion Time - 5 to 10 minutes

LABORATORY CONTROL PROCEDURE USED FOR H_2SO_4 - $Na_2Cr_2O_7$ DEOXIDIZER

Sulfuric Acid

1. Transfer 2 ml sample of solution to beaker. Add distilled water.
2. Add 5 drops of bromcresol green Indicator.
3. Titrate with standard NaOH solution to yellow-green or pH 5.
4. Calculation:

$$\text{gpl } H_2SO_4 = \text{ml NaOH} \times N \text{ NaOH} \times 24.5$$

5. Control at 22 to 28% by weight or 129 to 164 ml/liter. One milliliter

H_2SO_4 (DPM 961) contains 1.710 grams pure H_2SO_4 .

Sodium Dichromate

1. Transfer 5 ml sample into Iodine flask. Add distilled water.
2. Add 10 ml HCl.
3. Add 15 ml 0.9M KI solution.
4. Titrate with standard $Na_2S_2O_3$ solution using starch indicator.
5. Calculation:

$$\text{gpl } Na_2Cr_2O_7 \cdot 2H_2O = \text{ml } Na_2S_2O_3 \times N \text{ } Na_2S_2O_3 \times 9.93$$

6. Control at 2 to 3% by weight $Na_2Cr_2O_7$ or 20 to 30 gpl. One gram $Na_2Cr_2O_7$ requires 1.14 grams $Na_2Cr_2O_7 \cdot 2H_2O$ (DPM 956).

Solution Makeup

Used median concentration of each material (H_2SO_4 - $Na_2Cr_2O_7$) for test.

H_2SO_4 66°Be , at 25% by weight - 485.14 fl oz

$Na_2Cr_2O_7 \cdot 2H_2O$ - 84.3 oz/gal

Deionized Water - Remainder

BELL HELICOPTER DEOXIDIZER SOLUTION ANALYSIS RECORD

Date	Conc H_2SO_4 gram/liter	Conc $Na_2Cr_2O_7 \cdot H_2O$ grams/liter	Temp °F
3-18-75	244	36.1	72
3-24-75	240	35.9	-

b. McDonnell Douglas (MDC) per P.S. 12050.1

Laboratory tank size and solution volume are the same as used for the 4215 alkaline cleaner. (See Appendix A.1.)

Solution Concentration and Range Operating Conditions

HNO_3 (O-N-350) - 9.3-10.5 gals/100 gals (11.9-13.4 fl oz/gal)

CrO_3 (O-C-303) - 38-42 lbs/10 gals (6.1-6.7 opg)

HF (O-H-795) - 0.85-1.0 gal/100 gals (1.1-1.28 opg)

Temperature - Ambient

Tap Water - Remainder

Metal Removal Rate - 0.0001-0.0003 in/surface/10 minutes

LABORATORY CONTROL PROCEDURE USED FOR 1051 ETCH SOLUTION

Chromic Acid

1. Transfer one milliliter of sample to iodine flask containing distilled water.
2. Add 10 ml 3.6M H_2SO_4 .
3. Add 10 ml 0.9M KI.
4. Titrate with standard $Na_2S_2O_3$ solution, using starch as indicator.
5. Calculation:

$$\text{opg } CrO_3 = \text{ml } Na_2S_2O_3 \times N \text{ } Na_2S_2O_3 \times 4.47$$

Hydrofluoric Acid

1. Transfer one milliliter of sample to beaker containing distilled water.
2. Neutralize with NaOH to pH 7.0 to 7.5.
3. Add 0.5M AgNO₃ dropwise with stirring until dark brick red color develops; add 5 drops more.
4. Filter through Whatman No. 42 paper. Wash ppt several times with distilled water. Collect filtrate and all washings.
5. Add one drop 0.5M AgNO₃ to check filtrate.
6. Add 15 drops of alizarine red "S" indicator. Add 0.32M HNO₃ dropwise to develop light yellow color.
7. Add 2.5 ml of monochloroacetic acid buffer.
8. Titrate with standard Th(NO₃)₄·4H₂O solution just to disappearance of yellow color. Recommend use blank with yellow color.
9. Calculation:

$$\text{fl oz HF/gallon} = \text{ml Th(NO}_3)_4 \cdot 4\text{H}_2\text{O} \times \text{N Th(NO}_3)_4 \cdot 4\text{H}_2\text{O} \times 2.909$$

Nitric Acid

1. Transfer 5 ml sample to beaker containing distilled water.
2. Titrate with standard NaOH solution to pH 3.7.
3. Calculation:

$$\text{fl oz/gallon HNO}_3 =$$

$$[\text{ml NaOH} \times \text{N NaOH}] - [(1.39 \times \text{fl oz/gal HF}) + (0.075 \times \text{oz/gal CrO}_3)] \times 1.46$$

Metal Removal Rate

1. Use the three designated 2 x 4 x 0.050 inch panels of nonclad 6061-T6 alloy which have been alkaline cleaned, deoxidized, dried and weighed.
2. Immerse panels in the 1051 etch solution for 10 minutes at ambient temperature.
3. Rinse thoroughly. Use distilled water as a final rinse.
4. Dry in oven for 20 to 30 minutes at 200° to 220°F. Cool in desiccator. Reweigh.

5. Calculations:

- Determine the average surface area of the panels.
- Determine the average weight loss of the panels.
- Determine the weight loss for 2 x 4 inches significant surface area by:

$$\text{weight loss} = \frac{8 \times \text{weight loss of panel per 10 minutes}}{\text{average panel surface area}}$$

- Determine metal removal rate by:

$$\frac{\frac{\text{weight loss for } 8 \text{ in}^2}{8} \times 0.155}{\text{specific gravity of nonclad 6061 alloy} \times 2.54} = \text{thickness in inches}$$

Specific gravity = 2.70

Average surface area of 2 x 4 panel = 16.8049 in²

Solution Makeup
MDC 1051

HNO₃ (O-N-350) - 265.85 fl oz

CrO₃ (O-C-303) - 122.9 oz

HF (O-H-795) - 25.8 fl oz

MDC DEOXIDIZER SOLUTION (1051 ETCH) ANALYSIS RECORD

Date	HNO ₃ fl oz/gal	CrO ₃ ooz	HF fl oz/gal	Temp °F	Metal Removal Rate in/sur/10 min	Additions
3-17-75	13.36	6.95	1.298	72	0.0003	
4-23-75	12.92	6.86	1.13	-	-	7 fl oz HF
5-16-75	-	-	-	-	0.0001	20 fl oz HF
4-23-75	-	-	-	73	0.0001	
4-30-75	-	-	-	-	-	New Solution
5-05-75	-	-	-	-	0.0003	
5-12-75	-	6.49	-	-	-	100 grams CrO ₃
5-15-75	-	-	1.36	-	-	
5-15-75	12.40	-	-	-	0.0002	20 fl oz HNO ₃

c. Boeing Deoxidizer for Phosphoric Acid Anodize per BAC5555

Lab Tank Size

Tank size and solution volume are the same as used for the 4215 alkaline cleaner. (See Appendix A.1.)

Solution Concentration Range & Operating Conditions

HNO_3 (O-N-350) - 10-20 fl oz/gal

Deoxidizer Makeup 6 - 5.1-11.5 fl oz/gal (See Note)
(Amchem Prod., Inc.)

NOTE: Maintain with Replenisher No. 16

Temperature - 65°-80°F (ambient)

Metal Removal Rate - 0.00015 to 0.0004 Inch/surface/hour

LABORATORY CONTROL PROCEDURE USED FOR AMCHEM DEOXIDIZER NO. 6-16

Nitric Acid

1. Transfer 10 ml sample of deoxidizer solution to 50 ml volumetric flask.
Dilute to mark with distilled water.
2. Transfer 10 ml sample from this dilution to beaker. Add distilled water.
3. Titrate with 0.857N NaOH to pH 3.7.
4. Calculation:

For each ml titer less than 3.9 ml, add 3.28 fl oz/gal of HNO_3 .

Deoxidizer

1. Transfer 5 ml sample of deoxidizer solution to iodine flask. Add distilled water.
2. Add 10 to 15 ml of HCl.
3. Add 10 to 15 ml of 0.9M KI solution.
4. Titrate with standard 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ solution, using starch as indicator.
5. Calculation:

For each ml titer less than 15 ml, add 0.43 fl oz/gal of AMCHEM Deoxidizer Replenisher No. 16.

Metal Removal Rate

1. Use the three designated 2 x 3 x 0.032 inch panels of alclad 2024-T3 alloy* which have been alkaline cleaned, deoxidized, dried and weighed.
2. Immerse panels in the deoxidizer for 15 minutes at ambient temperature.
3. Rinse thoroughly. Use distilled water as a final rinse.
4. Dry in oven for 20 to 30 minutes at 200° to 220°F. Cool in desiccator. Reweigh.
5. Calculations:
 - (a) Determine the average surface area of the panels.
 - (b) Determine the average weight loss of the panels.
Multiply x 4 = weight loss per hour.
 - (c) Determine weight loss for 2 x 3 inches significant surface area by:

$$\text{weight loss} = \frac{6 \times \text{weight loss per hour}}{\text{average panel surface area}}$$

- (d) Determine metal removal rate by:

$$\frac{\frac{\text{weight loss for } 6 \text{ in}^2}{6} \times 0.155}{\text{specific gravity of alclad} \times 2.54} = \text{thickness in inches}$$

Average surface area of panel = 12.32 in².

Specific gravity for 7072 alclad = 2.72.

*Material changed to alclad 7075 alloy.

SOLUTION MAKEUP
AMCHEM NO. 6-16

HNO₃ (O-N-350) - 253.9 fl oz

Amchem No. 6 - 178.5 fl oz

Boeing BAC5555 Deoxidizer Solution Analysis Record

Date	MI Titer HNO ₃	MI Titer Deoxidizer	Temp °F	Metal Removal In/sur/hrs	Additions
3-04-75	4.4	25.4	72	0.00034	
3-07-75	4.5	22.8	74	-	
3-14-75	-	-	70	0.000044	32 fl oz No. 16
3-21-75	4.5	30.1	-	-	
4-18-75	4.4	25.0	73	0.00014	48 fl oz No. 16
4-30-75	4.1	30.4	-	-	
5-19-75	-	-	-	0.0004	
5-23-75	4.3	27.6	-	-	
5-30-75	-	-	-	0.00029	29 fl oz No. 16
6-04-75	3.5	22.9	74	-	500 ml HNO ₃
6-10-75	3.35	18.4	75	-	700 ml HNO ₃
6-12-75	-	-	75	0.00019	50 fl oz No. 16
6-20-75	2.3	11.85	75	-	Partial Dump - 3100 ml
6-23-75	-	-	73	0.00025	HNO ₃ , 32 fl oz No. 16
6-30-75	-	-	-	-	New Solution
7-02-75	3.7	20.7	75	0.00029	
7-09-75	-	-	75	0.00013	
7-10-75	3.4	16.7	75	-	300 ml HNO ₃ , 33 fl oz No. 16
7-15-75	-	-	-	0.00018	64 fl oz No. 16

APPENDIX "B"

PHOSPHORIC ACID ANODIZE

1. Phosphoric Acid Anodizing Solution Concentration Range and Operating

Conditions:

Phosphoric Acid (0-0-670, Class I) 85% H_3PO_4 -	11 to 16 oz/gal
Deionized Water	Remainder
Temperature	65°F to 90°F
Voltage (D. C.)	8 to 12 volts
Anodizing Time	20 to 25 minutes
Tank Lining	Chem Lead
Tank Size	10.25" wide 21" Long x 25" deep
Solution Volume	22.13 gal

2. Laboratory Control Procedure used for Phosphoric Acid Anodize Solution:

Phosphoric Acid (as primary H^+)

1. Transfer 10 ml sample of anodizing solution into beaker. Add distilled water.
2. Titrate with standard NaOH solution to pH 4.2
3. Calculation:

$$\text{opg } H_3PO_4 = \text{ml NaOH} \times N \text{ NaOH} \times 1.31$$

USE FOLLOWING PROCEDURE WHEN DISSOLVED ALUMINUM IN SOLUTION INTERFERES WITH TITRATION:

Phosphoric Acid (as two H^+)

1. Transfer 10 ml sample of anodizing solution into beaker. Add distilled water.
2. Add 5 ml of 5.3M $KF \cdot 2H_2O$ solution.
3. Titrate with standard NaOH solution to pH 8
4. Calculation:

$$\text{opg } H_3PO_4 = \text{ml NaOH} \times N \text{ NaOH} \times 0.66$$

3. Phosphoric Acid Solution Maintenance

Date	H ₃ PO ₄ opg	Temp °F	Additions
2-27-75	13.2	75	3 fl. oz. H ₃ PO ₄ rgr. grd.
3-4-75	12.6	75	19 fl. oz. H ₃ PO ₄
3-6-75	10.9	68	Diluted for opt. test
3-10-75	10.3	65	14 fl. oz. H ₃ PO ₄ rgt. grd.
3-10-75	10.37	77	56 fl. oz. H ₃ PO ₄ DPM 951
3-11-75	15.5	64	10 fl. oz H ₃ PO ₄ DPM 951
3-12-75	16.4	89	
3-21-75	14.5	76	
3-21-75	13.3		3.7 fl. oz. H ₃ PO ₄ rgt.
3-21-75	13.58		
4-2-75	13.7		
4-7-75	11.84		750 ml DPM 951; diluted H ₂ O @ 14.7 opg
4-8-75	13.50		
4-18-75	13.34	77	
4-30-75	12.29	67	100 ml H ₃ PO ₄ rgt.
5-15-75			New Solution
5-16-75	18.46	70	
5-19-75	13.53		New Solution
5-29-75	13.39	77	70 ml H ₃ PO ₄ rgt.
6-2-75	12.98	77.5	Filtered; 325 ml H ₃ PO ₄ rgt.
6-3-75	13.79	77	
6-5-75	13.10		Filtered; 200 ml H ₃ PO ₄ rgt.
6-12-75	12.95	75	Filtered; 300 ml H ₃ PO ₄ DPM
6-23-75	13.04		Filtered; 185 ml H ₃ PO ₄ rgt.
7-2-75	12.88		350 ml H ₃ PO ₄
7-3-75	13.24	77	Filtered; 115 ml H ₃ PO ₄ rgt.
7-10-75	13.17		Filtered; 150 ml H ₃ PO ₄
7-14-75	13.17	280	Filtered; 150 ml H ₃ PO ₄ rgt

4. Temperature and Humidity Study

a. On March 4, 1975, 8 panels 6x6x.125 inches 2024-T3 nonclad were identified as 2PI" and processed in a single load as follows:

- (1) Drilled 2 holes 1.26 inch diameter for racking along one edge.
- (2) Solvent cleaned each panel with chlorosolv solvent (DPM 5069).
- (3) Used aluminum wire 1100S, 0.125 inch for racking,
- (4) Alkaline cleaned panels in Turco 4215 for 15 minutes at 145-155°F.
- (5) Rinsed in hot tap water (156°F) for 2 1/2 minutes followed by a cold tap water rinse (72°F) for 2 1/2 minutes.
- (6) Deoxidized in Amchem 6-16 for 15 minutes.
- (7) Rinsed in cold overflowing tap water followed by deionize water rinse with hose.
- (8) The panels were immersed in the phosphoric acid and voltage was turned on within one (1) minute.
- (9) The voltage was raised stepwise to exactly 10 volts D.C. A Simpson Elect. Co. Model 260 Voltmeter was interposed for fine control of voltage.
- (10) The temperature of the bath was maintained as close as possible to 77.5°F.
- (11) The panels were rinsed in overflowing tap water at 70°F for 15 minutes and followed by rinsing with deionized water from a hose.

- (12) After final rinse, panels were placed in oven at 150°F for 30 minutes to dry.

Data Recorded during Anodizing

H ₃ PO ₄ Concentration		13.5 oz/gal
Temp. at start	-	76.8°F
Temp. at finish	-	78.8°F
Volts	-	10 DC.
Amps.	-	19.9
Total surface area of panels	-	600 in. ²
Total surface area of panels plus wire	-	622 in. ²
Amps per foot square	-	4.61

Type of Gassing - Vigorous smoke-like

Appearance of panels - No visible change.

- b. A second set of eight (8) panels 6x6x0.125 inches 7075-T6 nonclad were identified as "7PI" and processed in a single load as follows:

- (1) The procedure was same as for the 2024-T3 alloy (See Appendix B.4.1.a-j.) except one additional hole was drilled on opposite end of panel to balance in racking.

Data Recorded During Anodizing

H ₃ PO ₄ Concentration	-	13.5
Temp. at start	-	77.0°F
Temp. at finish	-	78.0°F
Volts	-	10 D.C.
Amps.	-	13.4
Total surface area of panels plus wire	-	643 in. ²
ASF	-	3.0

Type of Gassing - Moderate smoke-like

Appearance of panels - No visible change

5. Thick Adherend - Fatigue Test Panels

a. On March 5, 1975 a set of 6 panels 6x6x0.500 inches 2024-T351 nonclad were identified as "2P2" and processed in a single load as follows:

- (1) The procedure was the same as described in Appendix B.4.1.a-j except no holes were drilled in panels for racking. The racking wire was placed on edges of panels.

Data Recorded During Anodizing (3/5/75)

H ₃ PO ₄ Concentration	-	13.5
Temp. at start	-	78°F
Temp. at finish	-	78.1°F
Volts	-	10 D.C.
Amps.	-	21 Amps.
Total Surface Area	-	746 in. ²
ASF	-	4.03
Type of Gassing	-	vigorous smoke-like
Appearance	-	No change

b. A second set of six (6) panels 6x7x0.500 inches 7075-T651 alloy nonclad were identified as "7P2" and processed in a single load as follows:

- (1) The procedure was same as for the 2024 T3 alloy described in B.4.1 a-j.

Data Recorded During Anodizing (3/5/75)

H ₃ PO ₄ Concentration	-	13.5
Temp. at start	-	76.7°F
Temp. at finish	-	77.3°F
Volts	-	10 DC
Amps	-	13.2 D.C.
Total surface Area	-	645 in. ²
ASF	-	2.95
Type of Gassing	-	Moderate smoke-like
Appearance	-	No visible change

APPENDIX "C"

DETAILED BONDING AND HUMIDITY TEST PROCEDURE

1. Primer Application

- a. The primer was applied within two hours after anodize drying.
- b. The primer was thoroughly shaken or stirred to ensure that all solids were in suspension.
- c. The primer was spray applied to each faying surface in a wet uniform cross coat ($\frac{1}{4}$). Spray application was accomplished by use of a DeVilbiss JGA spray gun with FX fluid tip and needle and #36 air cap, with air pressure of 45 ± 5 psi at the spray gun and the fluid needle full open.

2. Adhesive Bonding

After primer application specimens were air dried for a minimum of 30 minutes. Primer was then force dried in an air circulating oven for a period of 60 ± 5 minutes at a temperature of $255 \pm 5^\circ\text{F}$.

Adhesive film FM73 .045#/ft² was removed from 0°F storage and allowed to come to ambient temperature before removing sealed bag.

One layer of adhesive film was placed in each bondline.

Specimens were vacuum bagged on a 1/4" aluminum sheet with 1/4" aluminum plate on the upper surface to obtain a flat bonding surface and get a uniform heat up on both surfaces.

Specimens were cured in an autoclave at a temperature of $245 \pm 5^\circ\text{F}$ for 90 minutes at 40 psi. A heat up rate of 3 - 6°F per minute from ambient temperature to the curing range was maintained.

Vacuum bag was vented to atmospheric pressure during the cure cycle.

Pressure was maintained during the cool down to 150°F .

Primer and bonding data is shown in Table C.

3. Wedge Crack Humidity Tests

Specimens consist of two 6 X 6 X .125 inch aluminum plates which have been surface treated, adhesive primed, and cured with one layer of adhesive film per the specified process.

After bonding panels are submitted for nondestructive testing of the bonded area. Any discrepancies or variations in the panels are noted and compared after panel is disassembled.

Panel are cut into five (5) 1 X 6 inch specimens. Saw cut edges are polished to highlight the glue line.

Glue line thickness measurements are noted for each specimen and recorded. Tapered 1 X 6 X .125 inch aluminum wedges are driven into the bond line at one end of the specimen to a depth of one inch. Specimens are allowed to stabilize for approximately one hour at ambient temperature. At this point the initial crack length is measured and scribed on the specimen. Specimens are then placed into an environmental chamber controlled at a temperature of 140°F at a relative humidity of 95 - 100% for a period of 60 minutes. Specimens are then removed from the chamber and any additional growth to the crack length is measured and recorded. Specimens are then replaced in the chamber for an additional two hours under the same environmental exposures. After a total of 3 hours exposure time total crack length is measured and recorded. Specimens are then disassembled and all surfaces are visually inspected for the mode of failure. Also at this time a correlation of the nondestructive test results are made.

Failure modes are recorded as cohesive, adhesive and percentage of adhesive failure, and adhesive failure at edge only.

TABLE C
PRIMER - BONDING DATA

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M B-86 ROLL 326
2P1	0.10	564	3- 4-75	
7P1	0.10		3- 4-75	
2FPL1	0.10		3- 4-75	
7FPL1	0.10		3- 4-75	
2P2	0.20		3- 5-75	
7P2	0.20		3- 5-75	
2FPL2	0.20		3- 5-75	
7FPL2	0.20		3- 5-75	
2P3CIVIT1+1	0.28		3- 7-75	
2P3CIV2T1+1	0.22		3- 7-75	
2P3CIVIT1+2	0.20		3- 7-75	
7P3CIV2T1+2	0.22		3- 7-75	
2P3CIV2T1+1	0.23		3- 7-75	
2P3CIVIT2+2	0.25		3- 7-75	
2P3CIV2T2+2	0.20		3- 7-75	
7P3CIVIT1+1	0.20		3-10-75	
7P3CIVIT1+2	0.20		3-10-75	
7P3CIV2T1+2	0.20		3-10-75	
7P3CIVIT2+1	0.20		3-10-75	
7P3CIV2T2+2	0.20		3-10-75	
7P3CIV2T2+1	0.20		3-10-75	
7P3CIVIT2+2	0.25		3-10-75	
2PIR	0.20		3-10-75	
7PIR	0.20		3-10-75	
2P3C2VIT1+1	0.20		3-11-75	
2P3C2V2T1+1	0.20		3-11-75	
2FPL1-1	0.20		3-11-75	
2FPL1-2	0.20		3-11-75	
2FPL1-3	0.20		3-11-75	
2FPL1-4	0.20		3-11-75	
7FPL1-1	0.20		3-11-75	

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M B-86 ROLL 326
7FPL1-2	0.20	564	3-11-75	
7FPL1-3	0.20		3-11-75	
7FPL1-4	0.20		3-11-75	
2P3C2VIT1+2	0.20		3-11-75	
2P3C2V2T1+2	0.20		3-11-75	
2P3C2V2T2+1	0.20		3-12-75	
2P3C2VIT2+1	0.20		3-12-75	
2P3C2VIT2+2	0.20		3-12-75	
2P3C2V2T2+2	0.20		3-12-75	
7P3C2V2T1+1	0.20		3-11-75	
7P3C2VIT1+1	0.20		3-11-75	
7P3C2VIT1+2	0.20		3-11-75	
7P3C2VIT2+1	0.20		3-12-75	
7P3C2V2T2+1	0.20		3-12-75	
7P3C2V2+2+2	0.15		3-12-75	
7P3C2VIT2+2	0.10		3-12-75	
2FPL1-1	0.20		3-12-75	
7FPL1-1	0.10		3-12-75	
2M4	0.25		3-18-75	
7M4	0.20		3-18-75	
2B4	0.30		3-19-75	
7B4	0.25		3-19-75	
7P10LSRT	0.15		3-24-75	
7FPL2SS-2	0.20		3-24-75	
7FPL2LS	0.10		3-24-75	
7P10SS-2	0.10		3-24-75	
7P10LSSS	0.15		3-24-75	
7FPL2SS-1	0.12		3-24-75	
7P10SS-1	0.15		3-24-75	
7M1LSRT	0.25		3-24-75	
7M1LSSS	0.25		3-24-75	
7M1SS-2	0.20		3-24-75	
7M1SS-1	0.18		3-24-75	
7B1LSRT	0.20		3-25-75	
7B1LSSS	0.25		3-25-75	
7B12SS-2	0.30		3-25-75	
7B12SS-1	0.35		3-25-75	

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M B-86 ROLL 326
7M5SIC2T1+2 S/S	0.30	564	4- 7-75	
2M5SIC2T1+1 S/S	0.225		4- 7-75	
7M5SIC2T1+1 S/S	0.20		4- 7-75	
2M5SIC2T1+2 S/S	0.20		4- 7-75	
2M5SIC2T1+2	0.20		4- 7-75	
2M5SIC2T1+1	0.20		4- 7-75	
7M5SIC2T1+1	0.20		4- 7-75	
7M5SIC2T1+2	0.20		4- 7-75	
2M5SIC1T1+1	0.25		4- 8-75	
2M5SIC1T1+1 S/S	0.20		4- 8-75	
2M5SIC1T1+2	0.25		4- 8-75	
2M5SIC1T1+2 S/S	0.20		4- 8-75	
7M5SIC1T1+1	0.25		4- 8-75	
7M5SIC1T1+1 S/S	0.20		4- 8-75	
7M5SIC1T1+2	0.25		4- 8-75	
7M5SIC1T1+2 S/S	0.20		4- 8-75	
7P13	0.20		4- 8-75	
7M5SIC2T2+1	0.25		4- 9-75	
7M5SIC2T2+1 S/S	0.20		4- 9-75	
7M5SIC2T2+2	0.20		4- 9-75	
7M5SIC2T2+2 S/S	0.20		4- 9-75	
2M5SIC2T2+1	0.20		4- 9-75	
2M5SIC2T2+1 S/S	0.20		4- 9-75	
2M5SIC2T2+2	0.20		4- 9-75	
2M5SIC2T2+2 S/S	0.25		4- 9-75	
7M5SIC1T2+1	0.20		4-10-75	
7M5SIC1T2+1 S/S	0.20		4-10-75	
7M5SIC1T2+2	0.20		4-10-75	
7M5SIC1T2+2 S/S	0.20		4-10-75	
7M5SIC1T2+1G	0.20		4-10-75	
7M5SIC1T2+2G	0.15		4-10-75	
2M5SIC1T2+1	0.15		4-10-75	

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 PATCH NO.	DATE APPLIED	ADHESIVE FM73M 8-86 ROLL 326
2M5S1C1T2+1 S/S	0.20	564	4-10-75	
2M5S1C1T2+2	0.20		4-10-75	
2M5S1C1T2+2 S/S	0.20		4-10-75	
2M5S1C1T2+1G	0.25		4-10-75	
2M5S1C1T2+2G	0.20		4-10-75	
7M5S2C2T1+1	0.25		4-11-75	
7M5S2C2T1+1 S/S	0.20		4-11-75	
7M5S2C2T1+2	0.20		4-11-75	
7M5S2C2T1+2 S/S	0.20		4-11-75	
2M5S2C2T1+1	0.25		4-11-75	
2M5S2C2T1+1 S/S	0.25		4-11-75	
2M5S2C2T1+2	0.20		4-11-75	
2M5S2C2T1+2 S/S	0.25		4-11-75	
7M5S2C1T1+1	0.20		4-14-75	
7M5S2C1T1+1 S/S	0.20		4-14-75	
7M5S2C1T1+2	0.25		4-14-75	
7M5S2C1T1+2 S/S	0.30		4-14-75	
2M5S2C1T1+1	0.20		4-14-75	
2M5S2C1T1+1 S/S	0.25		4-14-75	
2M5S2C1T1+2	0.20		4-14-75	
2M5S2C1T1+2 S/S	0.25		4-14-75	
2M5S2C1T2+1	0.10		4-15-75	
2M5S2C1T2+1	0.10		4-15-75	
2M5S2C1T2+1 S/S	0.10		4-15-75	
2M5S2C1T2+2	0.10		4-15-75	
2M5S2C1T2+2	0.10		4-15-75	
2M5S2C1T2+2 S/S	0.10		4-15-75	
7M5S2C1T2+1	0.10		4-15-75	
7M5S2C1T2+1	0.10		4-15-75	
7M5S2C1T2+1 S/S	0.10		4-15-75	
7M5S2C1T2+2	0.10		4-15-75	
7M5S2C1T2+2	0.10		4-15-75	
7M5S2C1T2+2 S/S	0.10		4-15-75	

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M B-86 ROLL 326
2M5S2C2T2+1	0.30	564	4-16-75	
2M5S2C2T2+1	0.25		4-16-75	
2M5S2C2T2+1 S/S	0.30		4-16-75	
2M5S2C2T2+2	0.23		4-16-75	
2M5S2C2T2+2	0.20		4-16-75	
2M5S2C2T2+2 S/S	0.28		4-16-75	
2M5S2C2T2+1	0.22		4-16-75	
2M5S2C2T2+1	0.21		4-16-75	
2M5S2C2T2+1 S/S	0.25		4-16-75	
2M5S2C2T2+2	0.28		4-16-75	
2M5S2C2T2+2	0.25		4-16-75	
2M5S2C2T2+2 S/S	0.28		4-16-75	
2M5S1C1T2+1X	0.19		4-17-75	
2M5S1C1T2+1X	0.21		4-17-75	
2M5S1C1T2+1XC	0.20		4-17-75	
2M5S1C1T2+1XC	0.20		4-17-75	
2M5S1C1T2+2X	0.20		4-17-75	
2M5S1C1T2+2X	0.18		4-17-75	
2M5S1C1T2+2XC	0.18		4-17-75	
2M5S1C1T2+2XC	0.20		4-17-75	
2M5S1C2T2+1X	0.20		4-17-75	
2M5S1C2T2+1X	0.20		4-17-75	
2M5S1C2T2+2X	0.25		4-17-75	
2M5S1C2T2+2X	0.22		4-17-75	
30 PANELS	--		4-18-75	
7214	0.22		4-21-75	
7214	0.25		4-21-75	
607475-761P14	0.30		4-21-75	
607475-761P14	0.30		4-21-75	
607475-761P14	0.27		4-21-75	
607475-761P14	0.29		4-21-75	

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PPIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M B-86 ROLL 326	
2M5SIC1T2+1Y	0.20	564	4-24-75		
2M5SIC1T2+2Y	0.20		4-24-75		
7M5SIC1T2+1Y	0.20		4-24-75		
7M5SIC1T2+2Y	0.20		4-24-75		
2M5SIC2T2+1Y	0.20		4-24-75		
2M5SIC2T2+2Y	0.20		4-24-75		
7M5SIC2T2+1Y	0.20		4-24-75		
7M5SIC2T2+2Y	0.20		4-24-75		
2M5SIC1T1+1Y	0.25		4-25-75		
2M5SIC1T1+2Y	0.25		4-25-75		
7M5SIC1T1+1Y	0.17		4-25-75		
7M5SIC1T1+2Y	0.22		4-25-75		
2M5SIC2T1+1Y	0.22		4-25-75		
2M5SIC2T1+2Y	0.17		4-25-75		
7M5SIC2T1+1Y	0.20		4-25-75		
7M5SIC2T1+2Y	0.20		4-25-75		
2M6CIV1T1+2	0.18		5- 2-75		
2M6CIV1T1+1	0.18		5- 2-75		
2M6CIV2T1+1	0.18		5- 2-75		
2M6CIV2T1+2	0.15		5- 2-75		
7M6CIV1T1+1	0.15		5- 2-75		
2M6CIV1T1+1 S/S	0.15		5- 2-75		
7M6CIV1T1+1 S/S	0.15		5- 2-75		
7M6CIV1T1+2	0.22		5- 5-75		
7M6CIV2T1+1	0.18		5- 5-75		
7M6CIV2T1+2	0.20		5- 5-75		

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 HATCH NO.	DATE APPLIED	ADHESIVE FM73M	
				B-86	ROLL 326
2M6CIVIT2+1	0.20	564	5- 6-75		
2M6CIVIT2+2	0.25		5- 6-75		
2M6CIV2T2+1	0.20		5- 6-75		
2M6CIV2T2+2	0.22		5- 6-75		
2M6CIV2T2+2 S/S	0.20		5- 6-75		
7M6CIVIT2+1	0.20		5- 7-75		
7M6CIV2T2+2	0.25		5- 7-75		
7M6CIV2T2+1	0.20		5- 7-75		
7M6CIV2T2+2	0.20		5- 7-75		
7M6CIV2T2+2 S/S	0.20		5- 7-75		
2M6C2VIT1+1	0.20		5- 8-75		
2M6C2V2T1+1	0.20		5- 8-75		
2M6C2VIT1+2	0.225		5- 8-75		
2M6C2V2T1+2	0.20		5- 8-75		
2M6C2VIT1+1 S/S	0.250		5- 8-75		
7M6C2VIT1+2	0.20		5- 9-75		
7M6C2VIT1+1	0.20		5- 9-75		
7M6C2V2T1+1	0.20		5- 9-75		
7M6C2V2T1+2	0.225		5- 9-75		
7M6C2VIT1+1 S/S	0.20		5- 9-75		
2M6C2V2T2+2	0.20		5-12-75		
2M6C2VIT2+2	0.20		5-12-75		
2M6C2V2T2+1	0.25		5-12-75		
2M6C2VIT2+1	0.25		5-12-75		
2M6C2V2T2+2 S/S	0.25		5-12-75		

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M P-86 ROLL 326	
7M602V2T2+2 S/S	0.28	564	5-13-75		
7M602V2T2+1	0.25	↓	5-13-75		
7M602V1T2+2	0.28	↓	5-13-75		
7M602V2T2+2	0.30	↓	5-13-75		
7M602V1T2+1	0.28	↓	5-13-75		
7P15L	0.225	560	5-20-75		
7P15D1	0.300	↓	5-20-75		
7P15D2	0.300	↓	5-20-75		
7P15DA		NO PRIMER			
7M602V1T2+1X	0.20	560	5-21-75		
7M602V1T2+2X	0.125	↓	5-21-75		
7M602V2T2+1X	0.150	↓	5-21-75		
7M602V2T2+2X	0.150	↓	5-21-75		
Task 111					
7p 3 2a	0.2 - 0.3		5-29-75		
7p 3 2b	0.2 - 0.3		5-30-75		
Task 112					
7p	0.2 - 0.3		6- 2-75		
7FXXX	0.28		6- 2-75		
7P227	0.225	↓	6- 2-75		↓

TABLE C (Cont'd)

SPECIMEN NO.	THICKNESS AV. (MILS)	PRIMER BR-127 BATCH NO.	DATE APPLIED	ADHESIVE FM73M B-86 ROLL 326
Task 208A				
Napkin Rings, Large	0.15	560	6- 5-75	
Napkin Rings, Small	0.15		6- 5-75	
Spindle	0.15		6- 5-75	
Task 107				
P7475-T761	0.18		6- 5-75	
P7475-T731	0.20		6- 5-75	
P7475-T61	0.25		6- 5-75	
Task 216 & 217	0.28		6- 5-75	
	0.25			
Task 107				
P7075-T73	0.30		6- 5-75	
P7075-T76	0.30		6- 5-75	

APPENDIX "D"

Phosphoric Acid Anodizing Process Parameter Confirmation

1. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as "2P3CIVIT1+1" where:

C1 = 11 oz/gal H_3PO_4

C2 = 16 oz/gal H_3PO_4

V1 = 8 volts

V2 = 12 volts

T1 = 65°F

T2 = 90°F

t1 = 20 min.

t2 = 25°F

2 = 2024-T3 alloy

7 = 7075-T6 alloy

P = Phosphoric Acid

3 = Task 103

a. Processing Procedure

- (1) Small holes (.126 inch) were drilled along sides of panels for racking.
- (2) The panels were solvent cleaned with Chlorosolv solvent.
- (3) The panels were racked with 1100S, 0.125 inch diameter aluminum wire.
- (4) The panels were Alkaline cleaned for 15 minutes at 145°F - 155°F in Turco 4215 cleaner.
- (5) After cleaning the panels were rinsed in hot tap water by immersion for 2 1/2 minutes followed by cold tap water rinse for 2 1/2 minutes.
- (6) The panels were deoxidized in Amchem 6-16 for 15 minutes, temperature 75°F.
- (7) The panels were rinsed in cold overflowing tap water followed by a rinse with deionized water from a hose.
- (8) The panels were immersed in the phosphoric acid solution and the voltage was turned on within one (1) minute.

- (9) The voltage was raised stepwise to exactly 8 volts. A Simpson Elect. Co. Model 260 Voltmeter was interposed for fine control of voltage.
- (10) The temperature of the bath was maintained at the specified temperature.
- (11) The panels were anodized for the specified time.
- (12) The panels were rinsed in overflowing tap water followed by a deionized water rinse with a hose.

b. Data Recorded During Anodizing (3/7/75)

- | | |
|--|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.9 |
| (2) Temperature of bath (T1) | - 65.1°F |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t1) | - 20 minutes |
| (5) Total Surface Area | - 162 in. ² |
| (6) Amps. per square foot | - 2.48 |
| (7) Gassing moderate at 6 volts but tapers down considerable. Voltage stabilizes after 10 minutes. | |

2. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as RP3 CIV2T1+1.

- a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/7/75)

- | | |
|--|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.9 |
| (2) Temperature of bath (T1) | - 64.9°F |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t1) | - 20 min. |
| (5) Total surface area | - 162 in. ² |
| (6) Amps. per square foot | - 3.1. |
| (7) Gassing was the same as Appendix D. 1.b.(7). | |

3. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as 2P3 CIV1Ti+2.

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/7/75)

- | | | |
|--|---|----------------------|
| (1) Concentration of H_3PO_4 (C1) | - | 10.9 |
| (2) Temperature of Bath (T1) | - | 65°F |
| (3) Volts D.C. - (V1) | - | 8 |
| (4) Time of Anodizing (t2) | - | 25 minutes |
| (5) Total surface area | - | 162 in. ² |
| (6) Amps. per square foot | - | 2.4 |
| (7) Gassing was same as Appendix D.1.b.(7) | | |

4. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as 2P3 CIV2Ti+2.

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/7/75)

- | | | |
|--|---|----------------------|
| (1) Concentration of H_3PO_4 (C1) | - | 10.9 |
| (2) Temperature of Bath (T1) | - | 65°F |
| (3) Volts D.C. (V2) | - | 12 |
| (4) Time of Anodizing (t2) | - | 25 minutes |
| (5) Total surface area | - | 162 in. ² |
| (6) Amps per square foot | - | 2.3 |
| (7) Gassing was same as Appendix D.1.b.(7) | | |

6. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as 2P3 CIVIT2t1.
 - a. The processing procedure was the same as described in Appendix D.1.a.
 - b. Data Recorded During Anodizing (3/17/75)

(1) Concentration of H_3PO_4 (C1)	-	10.9
(2) Temperature of Bath (T2)	-	90°F
(3) Volts D.C. (V1)	-	8
(4) Time of Anodizing (t1)	-	20 min.
(5) Total surface area	-	162 in. ²
(6) Amps. per square foot	-	9
(7) Gassing same as before.		
6. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as 2P3 CIV2T2t1.
 - a. The processing procedure was the same as described in Appendix D.1.a.
 - b. Data Recorded During Anodizing (3/17/75)

(1) Concentration of H_3PO_4 (C1)	-	10.9
(2) Temperature of Bath (T2)	-	90.2
(3) Volts D.C. (V2)	-	12
(4) Time of Anodizing (t1)	-	20 minutes
(5) Total surface area	-	162 in. ²
(6) Amps. per square foot	-	8
(7) Gassing same as above.		
7. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as 2P3 CIVIT2t2.
 - a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/7/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.9 |
| (2) Temperature of Bath (T2) | - 90.2 |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total surface area | - 162 in. ² |
| (6) Amps per square foot | - 6.22 |
| (7) Gassing was same as before. | |

8. Two panels 6x6x0.125 inches 2024-T3 alloy nonclad were identified as 2P3 CIV2T2t2.

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/7/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.9 |
| (2) Temperature of Bath (T2) | - 90°F |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total surface area | - 162 in. ² |
| (6) Amps per square foot | - Not recorded |

9. Two panels 6x6x0.125 inches 7075-T6 alloy nonclad were identified as 7P3 CIV1T1t1, also added one (1) panel 0.5x6x0.125 of 7075-T6 identified as "SI" to be used for analytical studies.

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

- (1) Concentration of H_3PO_4 (C1) - 10.3
- (2) Temperature of Bath (T1) - 65°F
- (3) Volts DC (V1) - 8
- (4) Time of anodizing (t1) - 20 minutes
- (5) Total surface area - 166.9 in.²
- (6) Amps. per square foot - 1.64
- (7) Gassing moderate at about 6 volts, but then after 10 minutes of anodizing, gassing is slight.

10. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 CIV2Ti+1 and one panel 0.5x6x0.125 inches was identified as "S2".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

- (1) Concentration of H_3PO_4 (C1) - 10.3
- (2) Temperature of Bath (T1) - 65°F
- (3) Volts D.C. (V2) - 12
- (4) Time of Anodizing - 20 min.
- (5) Total surface area - 167 in.²
- (6) Amps. per square foot - 1.9
- (7) Gassing even with increased voltage was very moderate.

11. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 CIV1Ti+2 and one (1) panel 7075-T6 nonclad .5x6x0.125 inches was identified as "S3".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.3 oz/gal |
| (2) Temperature of Bath (T1) | - 65.1°F |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t2) | - 25 min. |
| (5) Total surface area | - 167 in. ² |
| (6) Amps. per square foot | - 1.72 |
| (7) Gassing was moderate. | |

12. Two panels 6x6x0.125 inches 7075-T6 nonclad wire identified as

7P3 CIV2T1t2 and one panel .5x6x0.125 inches was identified as "S4".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.37 |
| (2) Temperature of Bath (T1) | - 65°F |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t2) | - 25 min. |
| (5) Total surface area | - 167 in. ² |
| (6) Amps. per square foot | - 1.72 |

13. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as

7P3 CIV1t2t1 and one panel .5x6x0.125 inches was identified as "S5".

a. The processing procedure was as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.37 oz/gal |
| (2) Temperature of Bath (T2) | - 90°F |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t1) | - 20 min. |
| (5) Total surface area | - 167 in. ² |
| (6) Amps. per square inch | - 4.8 |

14. Two panels 6x6x0.125 inches 7075-T6 nonclad was identified as 7P3 CIV2T2t1 and one panel 0.5x6x0.125 inches was identified as "S6".

a. The processing procedure was as described in Appendix D.1.a.

b. Data Recorded During Anodizing 3/10/75

(1) Concentration of H_3PO_4 (C1)	- 10.3 oz/gal
(2) Temperature of Bath (T2)	- 90.2°F
(3) Volts D.C. (V2)	- 12
(4) Time of Anodizing	- 20 min.
(5) Total surface area	- 167 in. ²
(6) Amps. per square foot	- 5.26

15. Two panels 6x6x0.125 inches 7075-T6 nonclad was identified as 7P3 C1 T2t2 and one panel 0.5x6x0.125 inches 7075-T6 nonclad was identified as "S7".

a. The processing procedure was as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

(1) Concentration of H_3PO_4 (C1)	- 10.37 oz/gal
(2) Temperature of Bath (T2)	- 90°F
(3) Volts D.C. - (V1)	- 3
(4) Time of Anodizing - (t2)	- 25 min.
(5) Total surface area	- 167 in. ²
(6) Amps per square foot	- 4.5

16. Two panels 6x6x0.125 inches 7075-T6 nonclad was identified as 7P3 CIV2T2t2 and one panel 0.5x6x0.125 inches was identified as "S8".

a. The process procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/10/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C1) | - 10.37 oz/gal |
| (2) Temperature of Bath (T2) | - 90°F. |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t2) | - 25 min. |
| (5) Total surface area | - 167 in. ² |
| (6) Amps per square foot | - |

17. Two panels 6x6x0.125 inches nonclad 2024-T3 were identified as 2P3 C2VIT1+1 and one panel 2024-T3 nonclad 0.5x6x0.125 inches was identified as "S11".

a. The processing procedure was as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/11/75)

- | | |
|-------------------------------------|------------------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 oz/gal. |
| (2) Temperature of Bath (T1) | - 64.5°F. |
| (3) Volts DC (V1) | - 8 |
| (4) Time of Anodizing (t1) | - 20 minutes |
| (5) Total surface area | - 181 in. ² |
| (6) Amps. per square foot | - 2.5 |

18. Two panels 6x6x0.125 inches nonclad 2024-T3 were identified as 2P3 C2V2T1+1 and one (1) panel 2024-T3 nonclad 0.5x6x0.125 inches was identified as "S12".

a. The processing procedure same as described in Appendix D.1.A.

b. Data Recorded During Anodizing (3/11/75)

- | | |
|-------------------------------------|----------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 |
| (2) Temperature of Bath (T1) | - 64.5°F |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t1) | - 20 minutes |
| (5) Total surface area | - Not recorded |
| (6) Amps per sq. foot | - Not recorded |

19. Two panels 6x0x0.125 inches 2024-T3 nonclad were identified as 2P3 02V1T1t2 and one (1) 0.5x6x0.125 inches 2024-T3 nonclad was identified as "S13"

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing

- | | |
|-------------------------------------|----------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 oz/gal. |
| (2) Temperature of Bath (T1) | - 85°F. |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total surface area | - Not recorded |
| (6) Total Amps. drawn | - 3.4 |

20. Two panels 6x0x0.125 inches 2024-T3 nonclad were identified as 2P3 02V2T1t2 and one small panel as above was identified as "S14".

a. Process procedure was same as described in Appendix D.1.a.

b. Data Recorded During Anodizing

- | | |
|-------------------------------------|----------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 oz/gal. |
| (2) Temperature of Bath (T1) | - 64.5°F. |
| (3) Volts D.C. - (V2) | - 12 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total Amps. | - 4.2 |

21. Two panels 6x0x0.125 inches 7075-T6 nonclad were identified as 2P3 02V1T1t1 and a small panel as before, identified as "S15".

a. Processing procedure was same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/11/75)

- | | |
|-------------------------------------|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 oz/gal |
| (2) Temperature of Bath (T1) | - 64.5°F. |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t1) | - 20 minutes |
| (5) Total Amps. D.C. | - 2.2 |

22. Two panels 6x6x0.125 inch 7075-T6 nonclad were identified as 7P3 C2V2T1+1 and one small panel as before, identified as "S16".

a. Processing procedure same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/11/75)

- | | |
|-------------------------------------|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 oz/gal |
| (2) Temperature of Bath (T1) | - 64.5°F |
| (3) Volts D.C. - (V2) | - 12 |
| (4) Time of Anodizing (t1) | - 20 minutes |
| (5) Total Amps. | - 2.5 |
| (6) Amps. per square foot | - 2.15 |

23. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 C2V1T1+2, and one small panel was identified as "S17".

a. Processing procedure was same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/11/75)

- | | |
|---|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 15.5 oz/gal |
| (2) Temperature of Bath (T1) | - 65°F. |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total Amps. D.C. | - 2.2 |
| (6) Packed wire on one side of panels only. | |

Two panels 6x0x0.125 inches 7075-T6 nonclad wire identified as

7075-02V2T1+2.

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/11/75)

- | | | |
|-------------------------------------|---|-------------|
| (1) Concentration of H_3PO_4 (C2) | - | 15.5 oz/gal |
| (2) Temperature of bath (T1) | - | 84°F. |
| (3) Volts D.C. used (V2) | - | 12 |
| (4) Time of Anodizing (t2) | - | 25 minutes |
| (5) Total Amps. D.C. | - | 2.4 |
| (6) Amps. per square foot | - | 2.2 |

25. Two panels 6x0x0.125 inches 2024-T3 nonclad were identified as

2024-02V1T2+1 and one panel 0.5x0x0.125 inches was identified as "S18".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75).

- | | | |
|-------------------------------------|---|----------------------|
| (1) Concentration of H_3PO_4 (C2) | - | 16.4 oz/gal |
| (2) Temperature of bath (T1) | - | 84.6°F. |
| (3) Volts D.C. used (V2) | - | 8 |
| (4) Time of Anodizing (t2) | - | 20 minutes |
| (5) Total surface area | - | 168 in. ² |
| (6) Amps. per square foot | - | 8.22 |
| (7) Anodizing solution | - | |

26. Two panels 6x0x0.125 inches 2024-T3 nonclad were identified as

2024-02V1T2+1 and one panel 0.5x0x0.125 inches was identified as "S18".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75)

- | | |
|-------------------------------------|----------------|
| (1) Concentration of H_3PO_4 (C2) | - 16.4 oz/gal. |
| (2) Temperature of Bath (T2) | - 89.6°F. |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t1) | - 20 min. |
| (5) Total Amps. | - 12.4 |
| (6) Gassing | - Vigorous |

27. Two panels 6x6x0.125 Inches 2024-T3 nonclad were identified as 2P3C2V1T2t2 and one small panel 0.5x6x0.125 inches was identified as "S20".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing 3/12/75

- | | |
|-------------------------------------|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 16.4 oz/gal |
| (2) Temperature of Bath (T2) | - 90.4°F. |
| (3) Volts D.C. - (V1) | - 8 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total Amps. D.C. | - 9.4 |
| (6) Gassing - | - Vigorous |

28. Two panels 6x6x0.125 Inches 2024-T3 nonclad were identified as 2P3 C2V2T2t2 and one panel 0.5x6x0.125 inches was identified as "S21".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75)

- | | |
|-------------------------------------|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 16.4 oz/gal |
| (2) Temperature of Bath (T2) | - 89.8°F |
| (3) Volts (D.C.) (V2) | - 12 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total Amps. D.C. | - 12.2 |
| (6) Gassing | - Vigorous |

29. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 C2VIT2t1 and one panel 0.5x6x0.125 inches was identified as "S22".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75)

(1) Concentration of H_3PO_4 (C2)	- 16.4 oz/gal
(2) Temperature of Bath (T2)	- 90.4°F.
(3) Volts D.C. (V1)	- 8
(4) Time of Anodizing (t1)	- 20 min.
(5) Total Amps. D.C.	- 5.8
(6) Cassing	- Mild

30. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 C2V2T2t1 one panel same alloy 0.5x6x0.125 inches was identified as "S23".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75)

(1) Concentration of H_3PO_4 (C2)	- 16.4 oz/gal
(2) Temperature of Bath (T2)	- 90°F.
(3) Volts D.C. - (V1)	- 12
(4) Time of Anodizing (t1)	- 20 minutes
(5) Total Amps.	- 7.6
(6) Cassing	- Mild

31. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 C2V1T2t2 and one panel same alloy 0.5x6x0.125 inches was identified as "S24".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75)

- | | |
|-------------------------------------|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 16.4 oz/gal |
| (2) Temperature of Bath (T2) | - 90°F |
| (3) Volts D.C. (V1) | - 8 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total Amps. D.C. | - 6.8 |
| (6) Gassing | - Mild |

32. Two panels 6x6x0.125 inches 7075-T6 nonclad were identified as 7P3 C2V2T2t2 and one panel same alloy 0.5x6x0.125 inches was identified as "S25".

a. The processing procedure was the same as described in Appendix D.1.a.

b. Data Recorded During Anodizing (3/12/75)

- | | |
|-------------------------------------|---------------|
| (1) Concentration of H_3PO_4 (C2) | - 16.4 oz/gal |
| (2) Temperature of Bath (T2) | - 90.4°F. |
| (3) Volts D.C. (V2) | - 12 |
| (4) Time of Anodizing (t2) | - 25 minutes |
| (5) Total Amps. | - 7.6 |
| (6) Gassing | - Mild |

Effects of Dissolved Aluminum in H_3PO_4 Anodizing Solution Upon Titration for H_2O_2 Concentration (3/13/75)

1. There are no limits for dissolved aluminum specified in BAC 5555. The specification states that any method of analysis may be used as long as it is not subject to interference from dissolved aluminum.
2. After the H_3PO_4 anodizing had a fair amount of use, a 800 ml sample was taken from tank. Four solution samples were prepared for titration as follows:

SOLUTION NO.	INGREDIENTS
a.	10 ml of anodizing solution
b.	10 ml of anodizing solution plus 5 ml of neutral 5.3M KF .2H ₂ O
c.	10 ml of anodizing solution plus 0.025 grams aluminum powder (USP grade)
d.	10 ml of anodizing solution plus 15 ml of neutral 5.3M KF .2H ₂ O plus 0.25 grams aluminum powder.

3. Each of the prepared sample solutions were titrated with standard NaOH to pH 8, to a level where secondary H⁺ is neutralized. This pH is necessary as addition of KF increases pH of anodizing solution to pH 5 plus.

The results of the titration was as follows:

SOLUTION NO.	ML OF 0.9678N NaOH	APPEARANCE OF SAMPLE AFTER ANODIZING
a.	27.0	White cloudy suspension
b.	27.0	Clear, water white with white ppt. at bottom.
c.	26.5	Heavy white cloudy suspension with white ppt. at bottom.
d.	26.8	Clear, water white with white ppt. at bottom.

4. Conclusions

- a. KF additions, that is, to sample solutions A and b do complex aluminum and leaves solution clear.
- b. Aluminum if present in "appreciable" amounts can interfere with the titration as can be seen with sample c and d.
- c. Samples a and b do contain dissolved aluminum but not enough to have an effect on the titration.
- d. For Control and maintenance of solution, the procedure will be changed to add KF and to titrate to pH 8 with correction in factor as follows:

$$\text{grams/liter } H_3PO_4 (2H+) = \frac{\text{ml NaOH} \times N_{NaOH} \times (98/2/1000) \times 1000}{10}$$

$$= \text{ml NaOH} \times N_{NaOH} \times 4.9$$

$$\text{Oz/gal } H_3PO_4 (2H+) = \text{ml NaOH} \times N_{NaOH} \times 0.66$$

CRACK EXTENSION TEST DATA

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP (°F)	START LENGTH	AFTER 1 HR. AT TEMP.	AFTER 3 HRS. AT TEMP.	COMMENTS
7P3C1VIT1-1	3-17-75	.0050"	140	2.143	.045	N.C.	COHESIVE FAILURE
-2		.0048"		2.176	.050	N.C.	
-3		.0062"		2.162	.065	N.C.	
-4		.0058"		2.234	.030	N.C.	
-5		.0038"		2.203	.065	N.C.	
7P3C1VIT2-1		.0050"		2.190	.053	N.C.	
-2		.0070"		2.308	.026	N.C.	
-3		.0050"		2.186	.032	N.C.	
-4		.0070"		2.290	.015	N.C.	
-5		.0052"		2.273	.015	N.C.	
7P3C1VIT2-1		.0060"		2.147	.078	N.C.	
-2		.0070"		2.145	.010	.023	
-3		.0077"		2.190	.005	N.C.	
-4		.0069"		2.182	.016	.040	
-5		.0067"		2.170	.010	.045	
7P3C2VIT1-1		.0058"		2.21	.055	.100	
-2		.0073"		2.22	.050	N.C.	
-3		.0048"		2.23	.050	.080	
-4		.0070"		2.21	.045	N.C.	
-5		.0066"		2.20	.057	.090	
7P3C2VIT2-1		.0043"		2.206	.017	N.C.	
-2		.0050"		2.200	.020	N.C.	
-3		.0071"		2.198	.040	N.C.	
-4		.0067"		2.284	.020	N.C.	
-5		.0035"		2.209	.008	N.C.	
7P3C2VIT2-1		.0038"		2.249	.042	N.C.	
-2		.0055"		2.142	.045	N.C.	
-3		.0065"		2.204	.041	N.C.	
-4		.0060"		2.231	.021	N.C.	
-5		.0072"		2.268	.010	.040	

TABLE D
CRACK EXTENSION TEST DATA

SPECIMEN I. D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH	AFTER 1 HR. AT TEMP.	AFTER 3 HRS. AT TEMP.	COMMENTS
TP3C2V2T1t2-1	3-17-75	.0055"	140	2.228	.045	.075	COHESIVE FAILURE
-2		.0058"		2.160	.100	N.C.	
-3		.0057"		2.123	.090	N.C.	
-4		.0066"		2.153	.047	N.C.	
-5		.0076"		2.200	.065	N.C.	
TP3C2V2T1t1-1		.0042"		2.266	.060	N.C.	
-2		.0062"		2.130	.095	N.C.	
-3		.0070"		2.115	.092	N.C.	
-4		.0051"		2.190	.066	N.C.	
-5		.0065"		2.322	.008	N.C.	
TP3C2V2T2t1-1		.0041"		2.300	.045	N.C.	
-2		.0060"		2.250	.030	N.C.	
-3		.0070"		2.250	Ø	.028	
-4		.0062"		2.275	Ø	.030	
-5		.0040"		2.275	.030	N.C.	
TP3C2V2T1t2-1		.0050"		2.227	.040	N.C.	
-2		.0080"		2.240	.010	.040	
-3		.0078"		2.202	.005	N.C.	
-4		.0068"		2.223	.045	N.C.	
-5		.0061"		2.206	.046	N.C.	
TP3C2V2T2t2-1		.0049"		2.180	.100	N.C.	
-2		.0068"		2.155	.090	N.C.	
-3		.0067"		2.264	.075	N.C.	
-4		.0050"		2.215	.035	N.C.	
-5		.0070"		2.185	.080	N.C.	
TP3C1V2T1t1-1	3-14-75	.0080"		2.30	.033	N.C.	
-2		.0068"		2.25	.015	N.C.	
-3		.0057"		2.23	.020	.064	
-4		.0050"		2.37	.043	N.C.	
-5		.0012"		2.27	.045	N.C.	

CRACK EXTENSION TEST DATA

SPECIMEN I. D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH	AFTER 1 HR. AT TEMP.	AFTER 3 HRS. AT TEMP.	COMMENTS
7P3CIV2T2-1	3-14-75	.0050"	140	2.23	.063	.070	COHESIVE FAILURE
-2		.0053"		2.23	.039	.050	
-3		.0065"		2.24	.030	N.C.	
-4		.0073"		2.25	.027	N.C.	
-5		.0078"		2.24	.012	N.C.	
7P3CIV2T2-1		.0059"		2.23	.100	N.C.	
-2		.0073"		2.23	.075	.078	
-3		.0050"		2.13	.132	.155	
-4		.0077"		2.22	.017	N.C.	
-5		.0059"		2.32	.061	N.C.	
7P3CIV1T2-1		.0059"		2.17	.092	N.C.	
-2		.0067"		2.07	.112	.117	
-3		.0074"		2.17	.087	N.C.	
-4		.0066"		2.13	.023	.043	
-5		.0051"		2.23	.051	.056	
7P3CIV2T1-1		.0041"		2.22	.088	.094	
-2		.0057"		2.13	.076	N.C.	
-3		.0053"		2.24	.063	N.C.	
-4		.0033"		2.23	.155	N.C.	
-5		.0017"		2.21	.047	N.C.	
2P3CIV1T1-1	3-17-75	.0045"		2.24	0-	N.C.	
-2		.0053"		2.13	.047	N.C.	
-3		.0060"		2.18	.032	N.C.	
-4		.0051"		2.17	.013	N.C.	
-5		.0039"		2.14	.035	N.C.	
2P3CIV1T1-1		.0048"		2.22	.025	N.C.	
-2		.0040"		2.16	.025	N.C.	
-3		.0055"		2.19	.045	N.C.	
-4		.0044"		2.21	.032	N.C.	
-5		.0055"		2.17	.009	N.C.	


TABLE D
CRACK EXTENSION TEST DATA

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP (°F)	START LENGTH	AFTER 1 HR. AT TEMP.	AFTER 3 HRS. AT TEMP.	COMMENTS
2P3C2VIT2-1	3-17-75	.0044"	140	2.20	.045	N.C.	COHESIVE FAILURE
-2		.0067"		2.14	.035	N.C.	
-3		.0058"		2.11	.015	N.C.	
-4		.0047"		2.20	.035	N.C.	
-5		.0057"		2.17	.040	N.C.	
2P3C2VIT1-1		.0035"		2.16	.036	N.C.	
-2		.0058"		2.15	.030	.055	
-3		.0068"		2.10	.020	N.C.	
-4		.0055"		2.12	.042	N.C.	
-5		.0034"		2.16	.035	N.C.	
2P3C2VIT1-1		.0039"		2.19	.060	N.C.	
-2		.0059"		2.18	.020	N.C.	
-3		.0035"		2.25	.010	N.C.	
-4		.0061"		2.20	Ø	.040	
-5		.0051"		2.24	.020	N.C.	
2P3C2VIT2-1		.0050"		2.19	.050	N.C.	
-2		.0065"		2.22	.047	N.C.	
-3		.0070"		2.22	.022	N.C.	
-4		.0062"		2.19	.030	.047	
-5		.0042"		2.27	.037	.060	
2P3C2V2T1-1		.0032"		2.27	.006	.018	
-2		.0043"		2.13	.018	N.C.	
-4		.0050"		2.19	Ø	N.C.	
-5		.0042"		2.20	.040	N.C.	
		.0049"		2.23	.030	N.C.	
2P3C2VIT2-1		.0048"		2.29	.020	N.C.	↑
-2		.0070"		2.28	.008	N.C.	
-3		.0052"		2.26	.046	N.C.	
-4		.0077"		2.22	.025	N.C.	
-5		.0068"		2.33	.006	N.C.	

TABLE D
CRACK EXTENSION TEST DATA

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP (°F)	START LENGTH	AFTER 1 HR. AT TEMP.	AFTER 3 HRS. AT TEMP.	COMMENTS
2P3C2V2T1t2-1	3-17-75	.0023"	140	2.12	.083	N.C.	COHESIVE FAILURE
-2		.0040"		2.17	Ø	N.C.	
-3		.0054"		2.12	Ø	N.C.	
-4		.0035"		2.18	.015	.035	
-5		.0053"		2.13	.020	N.C.	
2P3C2V2T2t1-1		.0070"		2.28	.009	N.C.	
-2		.0068"		2.19	.030	.042	
-3		.0077"		2.20	.022	N.C.	
-4		.0064"		2.19	.065	N.C.	
-5		.0072"		2.27	.042	N.C.	
2P3C2V2T2t2-1		.0050"		2.47	Ø	.030	
-2		.0063"		2.18	.050	N.C.	
-3		.0065"		2.15	.060	N.C.	
-4		.0052"		2.17	.080	N.C.	
-5		.0031"		2.30	.040	N.C.	

TABLE D
CRACK EXTENSION TEST DATA

SPECIMEN I.D.	DATE	GLUE LINE THICK.	TEST TEMP. (°F)	START LENGTH	AFTER 1 HR. AT TEMP.	AFTER 3 HRS. AT TEMP.	COMMENTS
2P3CIV2T2-1	3-17-75	.0041"	140	2.26	.080	N.C.	COHESIVE FAILURE
-2		.0055"		2.18	.094	N.C.	
-3		.0048"		2.23	.096	N.C.	
-4		.0050"		2.19	.052	N.C.	
-5		.0064"		2.20	.063	N.C.	
2P3CIV2T1-1		.0056"		2.19	.107	N.C.	 POROSITY POINTS
-2		.0053"		2.22	.053	N.C.	
-3		.0065"		2.20	.034	.050 *	
-4		.0065"		2.16	.048	N.C.	
-5		.0052"		2.27	.037	.042 *	
2P3CIV2T1-1		.0042"		2.19	.053	N.C. *	MEASUREMENT TAKEN AT END OF CRACK * POROSITY EXTENDED PAST CRACK EXTENSION POINT MEASURED AT END OF OPEN CRACK ONLY
-2		.0054"		2.15	.010	N.C.	
-3		.0055"		2.10	.086	N.C.	
-4		.0054"		2.23	.062	N.C.	
-5		.0049"		2.22	.030	N.C.	
2P3CIV2T1-1		.0039"		2.25	.090	N.C.	
-2		.0048"		2.19	.094	N.C.	
-3		.0037"		2.18	.042	.078	
-4		.0056"		2.20	.045	N.C.	
-5		.0053"		2.11	.105	N.C.	
2P3CIV2T2-1		.0046"		2.12	.060	N.C.	
-2		.0059"		2.15	.040	N.C.	
-3		.0096"		2.14	.082	N.C.	
-4		.0051"		2.06	.021	N.C.	
-5		.0058"		2.16	.058	N.C.	

APPENDIX "E"

CHROMIC ACID ANODIZE

1. Chromic Acid Anodizing Solution Concentration Range and Operating Conditions

(MDC per P.S. 13201, Rev. "J").

Chromic Acid (Free) (O-C-303)	6.0 - 8.0 oz./gal.
Total Acid	6.0 - 14.4 oz./gal.
Sulfates	4 oz./gal. max.
Chlorides	0.2 grams/liter max.
Alumina	10 grams/liter max.
pH	0.9 max.
Temperature	90° - 100°F
Water Deionized	Remainder

2. Chromic Acid Anodizing Solution Concentration Range and Operating Conditions

(Bell Helicopter BPS FW 4352, Rev. "G").

Chromic Acid (Free)	30 grams/liter min.
Total Acid	60 - 100 grams/liter
Sulfates	0.5 grams/liter max.
Chlorides	0.3 grams/liter max.
Alumina	10 grams/liter max.
pH	0.8 max.
Temperature	92° - 98°F
Water deionized	Remainder
Tank	Stainless steel
Tank Size	10" W. x 19.75" L. x 17.75" D.
Total Solution Volume	14.32 gal.

3. Laboratory Control Procedure Used for Both MDC and Bell Helicopter Chromic Acid Anodizing

Chromic Acid and Alumina

- a. Transfer 20 ml sample to beaker. Add distilled water.
- b. Titrate with standard 0.5N NaOH to pH 3.2 and record as titer "A".
- c. Continue titration to pH 4.8 and record titer "B".
- d. Calculation:

$$\text{mg free acid} = \text{titer "A"} \times 0.334$$

$$\text{mg total CrO}_3 = \text{titer "B"} \times 0.334$$

$$\text{mg alumina} = \text{titer "B"} \text{ less titer "A"} \times 0.47$$

Sulfates

- a. Transfer 20 ml sample to beaker. Add distilled water.
- b. Add 20 ml HAc.
- c. Add 5 ml HCl.
- d. Add 7 grams of hydroxylamine hydrochloride which has been dissolved in distilled water.
- e. Heat near boil. Allow to stand for 30 minutes.
- f. Warm and slowly add 10 ml of 0.41M BaCl₂ with constant stirring. Heat near boil.
- g. Allow solution to set overnight.
- h. Filter through Whatman No. 42.
- i. Wash ppt with distilled water. Wash ppt with acidified distilled water.
- j. Place ppt in weighed crucible and ignite to white ash @ 1300°F.
- k. Calculation:

For MDC, calculate BaSO₄ as H₂SO₄

$$\text{weight ppt} \times 21.01 = \text{mg H}_2\text{SO}_4$$

For Bell Helicopter, calculate BaSO₄ as SO₄

$$\text{weight ppt} \times 20.58 = \text{mg SO}_4$$

Chlorides

USE DISTILLED WATER THROUGHOUT FOR RINSING ALL GLASSWARE

- a. Transfer 25 ml of sample to beaker. Add distilled water.
- b. Add 10 ml of HNO_3 and heat to boiling.
- c. Slowly add 3 - 4 ml of 0.4M AgNO_3 with constant stirring.
- d. Digest for 1 - 2 hours at 140 - 176°F.
- e. Filter through weighed medium porosity sintered glass crucible.
- f. Wash with distilled water and dilute HNO_3 .
- g. Oven dry at 200 - 220°F to constant weight.
- h. Calculations:

For MDC, calculate AgCl as NaCl :

weight precipitate $\times 16.3 = \text{ppt chlorides as NaCl}$

For Bell Helicopter, calculate AgCl as chlorides:

weight precipitate $\times 9.88 = \text{ppt chlorides}$

4. Chromic Acid Anodize Solution Maintenance for the MDC Process

Date	Free Acid opg	Total Acid opg	Sulfates as gpl H_2SO_4	Chlorides as gpl NaCl	Alumina opl	pH	Temp. °F	Additions
3-17-75	6.78					0.81	80	
3-24-75	4.18	4.28			0.13			
3-24-75	7.16		0.00046			0.68	95	
4- 3-75	7.05	7.25			0.26	0.64	65	
4-10-75	6.48	6.98			0.65	0.79	95	Portion discarded
4-14-75	5.29					0.67	90	600g CrO_3
4-14-75	6.88	7.31		0.0021	0.56			
4-24-75	7.05	7.35			0.79	0.73	95	
5- 1-75	5.85	6.38			0.69	0.65		Conc lowered
5- 2-75	5.74	7.08			1.72	0.69	85	14g CrO_3
5- 6-75	5.64	6.38			0.95	0.81		108g CrO_3
5- 7-75	5.68	6.59		0.0049	1.18	0.65	100	
5- 7-75	8.02	8.82						970g CrO_3
5-12-75	7.98	8.88			1.16	0.61	100	
5-13-75				0.0285				
5-20-75	7.72	8.65			1.20	0.76		110g CrO_3
5-21-75			0.0010					

5. Chromic Acid Anodizing Solution Maintenance for the Bell Helicopter Process

Date	Free Acid opl	Total Acid opl	Sulfates opl	Chlorides opl	Alumina opl	pH	Temp. °F	Additions
3-18-75	65.85	67.13		0.0060	0.22	0.58	96	1300g CrO_3
3-24-75	54.08	55.58	0.0095		0.26	0.72	88	

6. Seal Solution Concentration Range and Operating Condition for MDC Chromic

Acid Anodize

Potassium Dichromate ($K_2Cr_2O_7$) 40 - 60 grams/liter
Tech., Harshaw Chem. Corp.

Specification: Appearance Orange-red crystals
Assay 99.8% min.
Chlorides as Cl .06% max.
Sulfates as SO_4 0.01% max.

Deionized Water Remainder

Tank Lining Fiberglass

Tank Size 9.75" W x 19" L. x 25.75" D.

Temperature 185 - 205°F

7. Laboratory Control Procedure for MDC Dichromate Anodic Sealer

Potassium Dichromate

- Obtain sample and allow to cool.
- Transfer 2 ml sample to iodine flask.
- Add 10 ml of 3.6M H_2SO_4 .
- Add 10 ml of 0.9M KI.
- Titrate with standard $Na_2S_2O_3$ solution, using starch indicator.
- Calculation:

$$gpl K_2Cr_2O_7 = ml Na_2S_2O_3 \times N Na_2S_2O_3 \times 24.5$$

8. Laboratory Control Procedure for Bell Helicopter Chromic Acid Sealer

Chromic Acid

- Obtain sample and allow to cool.
- Transfer 100 ml sample to iodine flask.
- Add 10 ml 3.6M H_2SO_4 .
- Add 10 ml 0.9M KI.
- Titrate with standard $Na_2S_2O_3$ solution, using starch indicator.

f. Calculation:

$$\text{gpl CrO}_3 = \text{ml Na}_2\text{S}_2\text{O}_3 \times N \text{ Na}_2\text{S}_2\text{O}_3 \times 0.33$$

$$\text{ppm CrO}_3 = \text{gpl CrO}_3 \div 0.001$$

g. Maintain pH 2.5 - 3.8 as measured at 25°C.

9. MDC Dichromate Sealer Solution Maintenance Record (Water Deionized and as Noted)

Date	K ₂ Cr ₂ O ₇ gpl	pH	Temp. °F	Additions
2-21-75	46.2	3.87	70	KOH 0.5g
3-18-75		4.32	197	KOH 1.0g
3-24-75		4.28	196	
3-27-75	43.8	4.39	68	182g K ₂ Cr ₂ O ₇
3-27-75	47.9	4.27		15g KOH
4- 3-75	40.5			1470g K ₂ Cr ₂ O ₇
4- 4-75	59.42	5.81		115g KOH
4- 9-75	63.04	5.86		Dumped for tap H ₂ O make-up
4-16-75	59.67	3.73		New solution
4-17-75		3.82	190	
4-23-75	58.38	3.98	130	
4-24-75		4.03	185	
4-24-75	63.05	4.03	210	After use

MDC Dichromate Anodic Sealer
(for Sealing Soln. Optimization)

Make-up: Use deionized water
Concentration @ 25 gpl
pH 4.2
Temp. 185 - 205°F

Date	$K_2Cr_2O_7$ gpl	pH	Temp. °F	Additions
4- 4-75	25.43	3.83	110	3g KOH
4- 4-75	25.17	4.28		
4- 7-75	25.17	4.43	150	NS as film on tank soln.
4-16-75	25.17	3.83		New solution
4-17-75		3.93	160	
4-23-75	24.39	4.12	130	
4-24-75		3.98	190	
4-24-75	25.04	3.98	148	After use
5- 1-75	49.04	3.91	162	Conc increased Task 6
5- 2-75	49.82	4.02	190	After use
5- 5-75		4.02	190	
5- 6-75	50.07		190	
5- 7-75		4.01	190	
5- 8-75	48.13	4.10	190	100g $K_2Cr_2O_7$
5- 9-75		4.14	190	
5-12-75		4.06	190	
5-13-75	49.95		190	
5-21-75		4.22	190	

MDC Dichromate Anodic Sealer
(for Sealing Soln. Optimization)

Make-up: Use tap water
Concentration @ 60 gpl
pH 6
Temp. 185 - 205°F

Date	K ₂ Cr ₂ O ₇ gpl	pH	Temp. °F	Additions
4-10-75	59.11	5.61		85g KOH; allow heat-disv
4-10-75	62.79			Dilute
4-11-75	60.19	5.77		25g KOH
4-15-75	59.29	5.82	150	
4-16-75		5.74	178	

MDC Dichromate Anodic Sealer
(for Sealing Soln. Optimization)

Make-up: Use tap water
Concentration @ 25 gpl
pH 6
Temp. 185 - 205°F

Date	K ₂ Cr ₂ O ₇ gpl	pH	Temp. °F	Additions
4-11-75	25.56	5.68		41g KOH
4-14-75	25.43	5.75	126	6.5g KOH
4-15-75		5.78	180	

10. Seal Solution Concentration Range and Operating Conditions for Bell

Helicopter Chromic Acid Anodize

Chromic Acid (CrO ₃) (O-C-303)	0.075 - 0.120 grams/liter
Deionized Water	Remainder
Temperature	180 - 185°F
Tank Lining	9.75" W. x 19" L. x 25.7" D.
Tank Volume	19.84 gal.

11. Bell Helicopter Sealer Solution Maintenance Record

Date	CrO ₃ ppm	pH	Temp. °F	Additions
2-21-75	108	3.03	70	
3-18-75		2.52	140	
3-24-75	101.3	3.05		

12. Chromic Acid Anodize Tradeoff Study - MDC versus Bell

a. MDC - Chromic Acid Anodize

- (1) Two panels 6 x 6 x 0.125 inches 2024-T3 nonclad were identified as "2M4" and one panel 0.5 x 6 x 0.125 inches same alloy was identified as "S26". The three panels were processed using the following procedure:
 - (a) The panels were solvent cleaned with chlorosolv solvent (DPM 5069).
 - (b) The panels were racked with 1100S, 0.125 inch diameter aluminum wire along one edge of panels.
 - (c) The panels were alkaline cleaned for 15 minutes at 145 - 155°F in Turco's 4215 cleaner.
 - (d) After cleaning the panels were rinsed in hot tap water by immersion for 2 1/2 minutes followed by cold tap water rinse for 2 1/2 minutes.
 - (e) The panels were deoxidized in 1051 etch for 10 minutes, temperature 72°F.
 - (f) The panels were rinsed in cold overflowing tap water.
 - (g) The panels were immersed in the chromic acid solution and the voltage was raised at a rate of 4 volts per minute to 20 volts. A Simpson Elect. Co., Model 260 Voltmeter was interposed for fine control of voltage.
 - (h) The temperature of the solution was maintained at the specified temperature.
 - (i) The panels were anodized for the specified time.

- (j) The panels were immersion rinsed for 2 1/2 minutes in cold tap water followed by an immersion rinse for 30 seconds to 60 seconds in deionized water and a cold deionized water rinse with a hose.
- (k) The panels were immersed in the seal solution at the temperature and time specified.
- (l) The panels were immersion rinsed for 2 1/2 minutes in deionized water, followed by a hose rinse with deionized water.
- (m) The panels were dried in oven at 150°F for 30 minutes.

Data Recorded during Anodizing and Sealing (3-18-75)

- | | |
|---------------------------------------|---------------------|
| (a) Temperature of Anodizing Solution | 94°F |
| (b) Voltage DC | 20 |
| (c) Time of Anodizing | 35 minutes |
| (d) Total Surface Area | 170 in ² |
| (e) Total Amps (DC) | 3.1 |
| (f) Amps per Square Foot | 2.62 |
| (g) Temperature of Seal Solution | 197°F |
| (h) Time in Seal Solution | 12 1/2 minutes |
- (2) Two panels 6 x 6 x 0.125 inches 7075-T6 nonclad were identified as "7M4" and one panel 0.5 x 6 x 0.125 inches same alloy was identified as "S27". The processing procedure was the same as before.

Data Recorded during Anodizing and Sealing

(a) Temperature of Anodizing Solution	95.5°F
(b) Voltage DC	20
(c) Time of Anodizing	35 minutes
(d) Amps per Square Foot	2.79
(e) Total Amps	3.3
(f) Temperature of Seal	197°F
(g) Time in Sealing Solution	12 1/2 minutes

b. Bell Helicopter Chromic Acid Anodize

- (1) Two panels 6 x 6 x 0.125 inches 2024-T3 were identified as "2B4" and one panel 0.5 x 6 x 0.125 inches was identified as "S2".

The three panels were processed using the following procedure:

- (a) The panels were solvent cleaned with chlorosolv (DPM 5069).
- (b) The panels were racked with 1100S, 0.125 inch diameter aluminum wire along one edge of panels.
- (c) The panels were alkaline cleaned for 15 minutes at 145 - 155°F in Turco's 4215 cleaner.
- (d) After cleaning the panels were rinsed in hot tap water by immersion for 2 1/2 minutes followed by cold tap water rinse for 2 1/2 minutes.
- (e) The panels were deoxidized in the Bell Helicopter deoxidizer for 7.5 minutes at 150°F.
- (f) The panels were immersion rinsed for 2 1/2 minutes in cold tap water followed by immersion rinse in deionized water for 30 seconds and a final rinse with deionized water from a hose.

- (g) The panels were immersed in the chromic acid solution and 5 - 10 volts were applied. Panels were anodized at this voltage for 2 - 2 1/2 minutes and then the voltage was increased 5 - 10 volts. The voltage was further increased at approximately one minute intervals to required voltage.
- (h) The panels after anodizing for the required time were rinsed in cold tap water for 2 1/2 minutes, immersion rinsed in deionized water for 30 seconds and final rinsed with deionized water from hose.
- (i) The panels were immersed in the Bell Helicopter seal solution at the temperature and time specified.
- (j) Without rinsing the panels were dried in an oven at 150°F for 30 minutes.

Data Recorded during Anodizing and Sealing (3-19-75)

(a) Temperature of Anodizing Solution	95°F
(b) Voltage DC	40
(c) Time of Anodizing	32.5 minutes
(d) Total Surface Area	170 in ²
(e) Total Amps	3.6
(f) Amps per Square Foot	3.05
(g) Temperature of Seal Solution	183°F
(h) Time In Seal Solution	8 minutes

- (2) Two panels 6 x 6 x 0.125 inches 7075-T6 nonclad were identified as "784" and one panel 0.5 x 6 x 0.125 inches was identified as "S29". The processing procedure was the same as before.

Data Recorded during Anodizing and Sealing (3-19-75)

(a) Temperature of Anodizing Solution	95°F
(b) Voltage DC	40
(c) Time of Anodizing	32.5 minutes
(d) Total Surface Area	170 in ²
(e) Total Amps	3.6
(f) Amps per Square Foot	3.05
(g) Temperature of Seal Solution	182°F
(h) Time in Seal Solution	8 minutes

APPENDIX "F"

CHROMIC ACID ANODIZE - SEALING PARAMETER CONFIRMATION

1. Three 6 x 6 x 0.125 Inches of 2024-T3 nonclad were identified as 2M5S1C2T1+2 where:

S1 = Deionized Water - pH 4.2

S2 = Tap Water - pH 6

C1 = $K_2Cr_2O_7$ at 25% by wt.

C2 = $K_2Cr_2O_7$ at 6% by wt.

T1 = Temp. 185°F

T2 = Temp. at 205°F

t1 = Time at 8 minutes

t2 = Time at 17 minutes

and one panel same alloy 0.5 x 6 x 0.125 Inches was identified as "S33".

This small panel will be used where necessary for instrumental analysis.

a. Processing Procedure

- (1) Three .126 inch diameter holes were drilled along sides of each panel for racking (two holes on one side and one hole on other).
- (2) The panels were cleaned with chlorosolv solvent.
- (3) The panels were racked with 1100S, 0.125 inch diameter aluminum wire.
- (4) The panels were Alkaline cleaned for 15 minutes at 145°F to 155°F in Turco's 4215 cleaner.
- (5) The panels were rinsed in hot tap water by immersion for 2 1/2 minutes followed by a cold tap water rinse for 2 1/2 minutes.
- (6) The panels were deoxidized in 1051 etch for 10 minutes, temperature ambient.
- (7) The panels were rinsed in cold overflowing tap water for 2 1/2 minutes followed by a rinse in deionized water for 30 seconds and then spray rinsed with deionized water from a hose.

- (8) The panels were then anodized in the MDC chromic acid process using the median range.
- (9) The panels after anodizing were rinsed cold tap water for 2 1/2 minutes, immersion rinsed in deionized water for 30 seconds, and rinsed with deionized water from hose.
- (10) The panels were sealed immediately at conditions specified.
- (11) One of the 6 x 6 x 0.125 inch panels was set aside for salt spray test.

b. Data Recorded During Sealing & Anodizing (4/7/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.94% by Wt.
(2) Type of Water for Seal (S1)	Deionized
(3) Temperature of Seal Soln (T1)	184-186°F
(4) Time in Seal solution (t2)	17 minutes
(5) pH of seal solution	5.81
(6) Temperature of Anodize solution	95°F
(7) Volts DC during anodizing	20
(8) Time of Anodizing	35 minutes
(9) Total surface area	254 in. ²
(10) Total Amps.	4.5
(11) Amps. per square foot	2.54

2. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2M51C2T1+1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S34".

a. The Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/7/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.94% by Wt.
(2) Type of water for Seal (S1)	DI
(3) Temperature of Seal Soln. (T1)	185°F

(4) Time in Seal soln (t1)	8 minutes
(5) pH of Seal soln	5.81
(6) Volts DC during anodizing	Waivered 18-20V
(7) Total Amps.	5.2

3. Three panels 6 x 6 x 0.125 Inches of 7075-T6 nonclad were identified as 705S1C2T1t1 and one panel 0.5 x 6 x 0.125 Inches of the same alloy was identified as "S38".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/7/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.94% by Wt.
(2) Type of water for seal (S1)	D1
(3) Temperature of Seal Soln. (T1)	185°F
(4) Time in Seal soln. (t1)	8
(5) pH of Seal soln.	5.81
(6) Volts D. C. during anodizing	(Voltage steady) 20
(7) Temperature of Anodize soln.	95°F
(8) Amps. per square foot	2.58

4. Three panels 6 x 6 x 0.125 Inches of 7075-T6 nonclad were identified as 705S1C2T1t2 and one panel 0.5 x 6 x 0.125 of the same alloy was identified as "S40".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/7/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.94% by Wt.
(2) Type of water for seal (S1)	D1
(3) Temperature of Seal Soln. (T1)	185°F
(4) Time in Seal soln. (t2)	17 minutes
(5) pH of Seal soln.	5.81
(6) Volts DC during anodizing	20
(7) Temperature of Anodize Soln.	94.4°F

5. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2155ICT1t1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S36".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/8/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.94% by wt.
(2) Type of water used for seal (S1)	D1
(3) Temperature of Seal Soln (T1)	185
(4) Time in Seal soln (t1)	8 minutes
(5) pH of Seal solution	5.81
(6) Total Amps. used	4.7
(7) Amps. per square foot	2.68

6. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2155ICT1t2 and one panel 0.5 x 6 x 0.125 inches of the same alloy was lost in processing tank.

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/8/75)

(1) Concentration of $K_2Cr_2O_7$ (C1)	2.517% by wt.
(2) Type of water for Seal Soln. (S1)	D1
(3) Temperature of Seal Soln (T1)	184°F-186°F
(4) Time in Seal soln.(t2)	17 minutes
(5) pH of Seal soln	4.45
(6) Total Surface Area	247.2 in. ²
(7) Total Amps. during Anodizing	4.5
(8) Amps. per square foot	2.00
(9) Temperature of Anodize Bath	95°F

7. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7MSICIT1t1 and one panel 0.5 x 6 x 0.125 inches was identified as "S37".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/8/75)

(1) Concentration of $K_2Cr_2O_7$ (Cl)	2.517% by Wt.
(2) Type of water for Seal Soln (SI)	DI
(3) Temperature of Seal Soln (TI)	184°F to 186°F
(4) Time In Seal soln (t1)	8 minutes
(5) pH of Seal soln	4.43
(6) Amps. per square foot	2.74
(7) Temperature of Anodizing soln.	94.5°F
(8) Total Amps. DC	4.8

8. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7MSICIT1t2 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S39".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/8/75)

(1) Concentration of $K_2Cr_2O_7$ (Cl)	2.517% by Wt.
(2) Type of water for Seal Soln. (SI)	DI
(3) Temperature of Seal Soln. (TI)	184°F to 186°F
(4) Time In Seal Soln (t2)	17 minutes
(5) pH of Seal soln	4.43
(6) Amps. per sq. ft.	2.74
(7) Total Amps. DC	4.8
(8) Temperature of Anodize Soln	94°F

9. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2M5S1C2T2+1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S35".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/9/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.3% by Wt.
(2) Type of water for Seal Soln. (S1)	D1
(3) Temperature of Seal Soln (t2)	205°F
(4) Time in Seal soln (t1)	8 minutes
(5) pH of seal soln.	5.86
(6) Amps. per square foot	2.74
(7) Total Amps used	4.8
(8) Temperature of Anodize Soln	94.2°F

10. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2M5S1C2T2+2 and one panel 0.5 x 6 x 0.125 inches of the same alloy is identified as "S41".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/9/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.3% by Wt.
(2) Type of water for Seal Soln (S1)	D1
(3) Temperature of Seal Soln (T2)	205°F
(4) Time in Seal soln (t2)	17 minutes
(5) pH of the Seal soln	5.86
(6) Amps. per square foot	Not recorded
(7) Total Amps. DC	4.8
(8) Temperature of Anodize soln	94.5°F

11. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5S1C2T2t1 and one panel 0.5 x 6 x 0.125 in same alloy was identified as "S42".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/9/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.3% by Wt.
(2) Type of water for Seal Soln (S1)	D1
(3) Temperature of Seal Soln (T2)	205°F
(4) Time in Seal soln (t1)	8 minutes
(5) pH of the Seal soln	5.86
(6) Amps. per square foot	2.79
(7) Total Amps. DC	4.9
(8) Temperature of Anodize soln	95°F

12. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5S1C2T2t2 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S43".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/9/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.3% by Wt.
(2) Type of water for Seal Soln (S1)	D1
(3) Temperature of Seal Soln (T2)	205°F
(4) Time in the Seal soln (t2)	17 minutes
(5) pH of the seal soln	5.86
(6) Total Amps. DC	4.9

13. Four 6 x 6 x 0.125 inch panels of 2024-T3 nonclad of which two were identified as 2M5SICIT2+1 and two were identified as 2M5SICIT2+1G. All panels identified with "G" were sealed in a 6 liter glass battery jar. One panel 0.5 x 6 x 0.125 inches was identified as "S44."

a. The processing procedure (cleaning and anodizing) was the same as described in Appendix F.1.a.

The anodizing was done at the medium range.

b. Data Recorded During Sealing and Anodizing (4/10/75)

(1) Concentration of $K_2Cr_2O_7$ in glass container (C1)	2.516% by Wt.
(2) Concentration of $K_2Cr_2O_7$ in plastic container (C1)	2.517
(3) Type of water for both Seal Solutions (S1)	D1
(4) Time in both seal solutions (t1)	8 minutes
(5) pH of seal solution glass	3.8
(6) pH of seal soln in plastic tank	3.9
(7) Temperature of both seal solutions	205°F
(8) Total surface area anodized	355.7 in. ²
(9) Amps. per square foot	2.59
(10) Total Amps. DC	6.4

14. Four panels 6 x 6 x 0.125 inches of 2024-T3 nonclad of which two were identified as 2M5SICIT2+2 and two were identified as 2M5SICIT2+2G (sealed in glass container). One panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S45".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/10/75)

(1) Concentration of $K_2Cr_2O_7$ (C1) in Glass	2.16%
(2) Concentration of $K_2Cr_2O_7$ (C1) in Plastic	2.517
(3) Type of water for seal solutions	D1
(4) Time in Seal Solutions (t2)	17 minutes
(5) pH of seal solutions in Plastic tank	3.9

(6) pH of seal solution in Glass tank	3.8
(7) Temperature of seal solutions	205°F
(8) Anodizing temperature	94.5°F
(9) Total Amps. DC	6.2

15. For 6 x 6 x 0.125 inches of 7075-T6 nonclad panels of which two were identified as 7M5SICIT2+1 and two were identified as 7M5SICIT2+1G (sealed in glass container) one panel 0.05 x 6 x 0.125 inches of the same alloy was identified as "S46".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/10/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1) in Glass	2.51% by wt.
(2) Concentration of the $K_2Cr_2O_7$ (C1 in Plastics	2.517% by wt.
(3) Type of water for seal solution (S1)	D1
(4) Time in seal solutions (+1)	8 minutes
(5) pH of seal solution in Plastic tank	3.9
(6) pH of seal solution in Glass tank	3.8
(7) Temperature of seal solution in Glass (T2)	204°F
(8) Temperature of seal solution in Plastic (T2)	203°F
(9) Anodizing temperature	94.5
(10) Total Amps. DC	6.4
(11) Amps. per square foot	2.89

16. Four panels 6 x 6 x 0.125 inches of 7075-T6 nonclad of which two were identified as 7M5SICIT2+2 and two were identified as 7M5SICIT2+2G (sealed in glass container). One panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S47".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/10/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1) in Glass	2.51% by Wt.
(2) Concentration of the $K_2Cr_2O_7$ (C1) in Plastic	2.517
(3) Type of water for seal solutions (S1)	H ₂ O
(4) Time in seal solutions (t2)	17 minutes
(5) pH of seal solution in plastics tank	3.9
(6) pH of seal solution in Glass container	3.8
(7) Temperature of seal solution in Glass tank	205°F
(8) Temperature of seal solution in Plastic tank	204°F
(9) Anodizing temperature	95°F
(10) Total Amps. DC	6.5

17. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad alloy were:

identified as 2M5S2C2T1t1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S48".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/11/75)

(1) Concentration of the $K_2Cr_2O_7$ (C2)	0.01% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t1)	8 minutes
(4) Temperature of seal solution (T1)	185°F
(5) pH of seal solution	5.77
(6) Temperature of Anodizing solution	94.5°F
(7) Total Amps. during anodizing	4.8
(8) Amps. per square foot	2.72
(9) Total surface area	253.8 in. ²

18. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 215S2C2T1+2 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S49".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/11/75)

(1) Concentration of the $K_2Cr_2O_7$ (C2)	6.01% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (+2)	17 minutes
(4) Temperature of the seal solution (T1)	185°F
(5) pH of seal solution	5.77
(6) Temperature of Anodizing Solution	95°F
(7) Total Amps. during anodizing	4.8
(8) Amps. per square foot	2.72
(9) Total surface area	253.8 in. ²

19. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 745S2C2T1+1 and one panel 0.5 x 6 x 0.125 inch of the same alloy was identified as "S50".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/11/75)

(1) Concentration of the $K_2Cr_2O_7$ (C2)	6.01% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (+1)	8 minutes
(4) Temperature of seal solution (T1)	185°F
(5) pH of seal solution	5.77
(6) Temperature of Anodizing solution	95°F
(7) Total Amps. during Anodizing	4.9
(8) Amps. per square foot	2.78
(9) Total surface area	253.8 in. ²

20. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 715S2C2T1t2 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S51".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/11/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.01% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t2)	17 minutes
(4) Temperature of seal solution (T1)	185°F
(5) pH of seal solution	5.77
(6) Temperature of Anodizing solution	95°F
(7) Total Amps. during anodizing	4.8
(8) Amps. per square foot	2.78
(9) Total surface area	253.8 in. ²

21. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 215S2C1T1t1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S52".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/14/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t1)	8 minutes
(4) Temperature of seal solution (T1)	185°F
(5) pH of seal solution	5.75
(6) Temperature of Anodizing Soln	94.5°F
(7) Total Amps. during anodizing	4.7
(8) Amps. per square foot	2.65
(9) Total surface area	255.6 in. ²

22. Three panels 6 x 6 x 0.125 inches of 2024T3 nonclad were identified as 2M5S2C1Ti12 and one panel 0.5 x 6 x 0.125 inch of the same alloy was identified as "S53". The small panel which is used for instrumental analysis was lost in the Alkaline Cleaner tank.

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/11/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t2)	17 minutes
(4) Temperature of seal solution (T1)	186°F
(5) pH of the seal solution	5.75
(6) Temperature of Anodizing solution	94.5°F
(7) Total Amps. during anodizing	4.7
(8) Amps. per square foot	2.65
(9) Total surface area	2.55.6 in. ²

23. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5S2C1Ti1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S54".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/14/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t1)	8 minutes
(4) Temperature of seal solution (T1)	183°F
(5) pH of seal solution	5.75
(6) Temperature of Anodizing soln	94.5°F
(7) Total Amps. during anodizing	4.8
(8) Amps. per square foot	2.70
(9) Total surface area	255.6 in. ²

24. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7MS2C1T1+2 and one panel 0.5 x 6 x 0.125 inch of the same alloy was identified as "S55".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/14/75)

(1) Concentration of $K_2Cr_2O_7$ (C1)	2.54% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t2)	17 minutes
(4) Temperature of seal solution (T1)	184°F
(5) pH of seal solution	5.75
(6) Temperature of Anodizing solution	94.5°F
(7) Total Amps. during anodizing	4.7
(8) Amps. per square foot	2.76
(9) Total surface area	255.6 in. ²

25. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad was identified as 7MS2C1T2+1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S56".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/15/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t1)	8 minutes
(4) Temperature of seal solution (T2)	205°F
(5) pH of seal solution	5.78
(6) Temperature of Anodizing solution	94.5°F
(7) Total Amps. during anodizing	4.6
(8) Amps. per square foot	2.59
(9) Total surface area	255.6 in. ²

26. Three panels same as above except changed time (t) in seal. Identified as 2M5S2CIT2t2, small panel identified as "S57".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/15/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Time in seal solution (t2)	17 minutes
(4) Temperature of Seal solution (T2)	205°F
(5) pH of seal solution	5.76
(6) Temperature of Anodize solution	94.5°F
(7) Total Amps. DC	4.6
(8) Total surface area	same as above
(9) Amps. per square foot	No change

27. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5S2CIT2t1 and one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S58".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/15/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by Wt.
(2) Type of water for seal (S2)	Tap
(3) Time in seal solution (t1)	8 minutes
(4) Temperature of seal solution (T2)	205.5°F
(5) pH of seal solution	5.78
(6) Temperature of Anodize solution	95°F
(7) Total Amps. DC	4.6
(8) Total surface area	No change
(9) Amps. per square foot	No change

28. Three panels, same as above, except changed time (t) in seal. Identified as 7M5S2C1T2t2, small panel identified as "S59".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/15/75)

(1) Concentration of the $K_2Cr_2O_7$ (C1)	2.54% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Temperature of seal solution (T2)	203°F
(4) Time in seal solution (t2)	17 minutes
(5) pH of seal solution	5.78
(6) Temperature of Anodizing solution	95.5°F
(7) Total Amps. DC	4.9
(8) Amps. per square foot	2.76
(9) Total surface area	same

29. Three panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2M5S2C2T2t1 and one panel 0.5 x 6 x .125 inches of the same alloy was identified as "S60".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/16/75)

(1) Concentration of the $K_2Cr_2O_7$ (C2)	5.93% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Temperature of seal solution (T2)	205°F
(4) Time in seal solution (t1)	8 minutes
(5) pH of seal solution	5.74
(6) Temperature of Anodizing solution	95°F
(7) Total Amps. DC	4.8
(8) Amps. per square foot	2.70
(9) Total surface area	255.6 in. ²

30. Three panels same as above except changed time (t) in seal. Identified as 2M5S2C2T2+2, small panel identified as "S61".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/16/75)

(1) Concentration of the $K_2Cr_2O_7$ (C2)	5.93% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Temperature of seal solution (T2)	204°F
(4) Time in seal solution (t2)	17 minutes
(5) pH of seal solution	5.74
(6) Temperature of Anodizing solution	94.5°F
(7) Total Amps. DC	4.8
(8) Amps. per square foot	same as above
(9) Total surface area	same as above

31. Three panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5S2C2T2+1 and one panel 0.5 x 6 x 0.125 inches was identified as "S62".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/16/75)

(1) Concentration of the $K_2Cr_2O_7$ (C2)	5.937% by Wt.
(2) Type of water for seal solution (S2)	Tap
(3) Temperature of seal solution (T2)	206°F
(4) Time in seal solution (t1)	8 minutes
(5) pH of seal solution	5.74
(6) Temperature of Anodizing solution	95°F
(7) Total Amps. DC	5.1
(8) Amps. per square foot	2.87
(9) Total surface area	255.6 in. ²

32. Three panels same as above except changed time (t) in seal solution.

Identified as 7M5S2C2T2t2, small panels identified as "S63".

a. The processing procedure was the same as described in Appendix F.I.a.

b. Data Recorded During Sealing & Anodizing (4/16/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.93% by wt.
(2) Type of water for seal solution (S2)	Tap
(3) Temperature of seal solution (T2)	205°F
(4) Time in seal solution (t2)	17 minutes
(5) pH of seal solution	5.74
(6) Temperature of Anodizing solution	95°F
(7) Total Amps. DC	4.8
(8) Amps. per square foot	same as above
(9) Total surface area	same as above

TAP WATER ANALYSIS

DATE SAMPLED	pH	TEMPERATURE	TOTAL SOLIDS
4/2/75	7.24	69°F	200 ppm
4/16/75	7.84	74°F	190 ppm

ADDITIONAL ANODIC SEALER SOLUTION CONFIRMATION TESTS

33. Adhesion failures from confirmation tests prompted following tests to pin point those conditions which may have caused and/or contributed to failures.

Solution Preparation

- a. Prepared a seal solution in a plastic wall tank at 2.5% by weight of $K_2Cr_2O_7$ in DI water. The pH = 3.90 (no adjustment).
 - b. Prepared in another plastic wall tank a seal solution of 6% by weight of $K_2Cr_2O_7$ in DI water. The pH = 3.80 (no adjustment).
 - c. Prepared a seal solution in an eleven (11) liter glass tank at 2.5% by weight of $K_2Cr_2O_7$ in DI water. The pH = 3.80 (no adjustment).
 - d. Two panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5SICIT2+IX plus a small panel 0.5 x 6 x 0.125 identified as "S64", and two panels same size and alloy were identified as 7M5SICIT2+IXG and a small panel identified as "S65".
- a. All the above panels were cleaned and anodized together using the processing procedure described in Appendix F.1.a.
 - b. Panels marked with "X" were sealed in the plastic wall tank for 8 minutes at 2.5% by weight of $K_2Cr_2O_7$.
 - c. Panels that were marked "XG" were sealed in the glass tank for eight (8) minutes.
 - d. Data Recorded During Sealing & Anodizing (4/17/75)

(1) Temperature of seal solution in plastic tank	204°F
(2) Temperature of seal solution in glass tank	205°F
(3) Temperature during Anodizing	94.5°F
(temperature went to 98°F)	
(4) Total surface area	335.6 in. ²
(5) Total Amps. DC	7.4

34. Four panels same as F.33, of which two were identified as 7M5SICIT2+2X and two were identified as 7M5SICIT2+2XG. Both sets were sealed for 17 minutes.

a. The processing procedure was the same as described in Appendix F.1.a.

b. Panels were sealed in solution according to their identification.

c. Data Recorded During Sealing & Anodizing (4/17/75)

(1) Temperature of seal solution in plastic tank	204°F
(2) Temperature of seal solution in Glass tank	203°F
(3) Temperature during Anodizing	96°F
(4) Total surface area	same
(5) Total Amps. DC	7.2

35. Two panels 6 x 6 x 0.125 inches of 7075-T6 nonclad identified as 7M5SICIT2+1X were sealed in a 6% by weight $K_2Cr_2O_7$ in the plastic wall tank at 205°F for 8 minutes. Two panels same as above, identified as 7M5SICIT2X were sealed in 6% $K_2Cr_2O_7$ in the plastic tank at 205°F for 17 minutes.

a. Data Recorded During Sealing & Anodizing (4/17/75)

(1) Temperature of seal solution	205°F
(2) Temperature of anodize solution	96°F
(3) Total Amps. DC	6.8

Since the results of the last series of test conducted ("X" and "XG") proved to be acceptable, this series of tests were conducted as a more inclusive confirmation of the initial series of tests.

36. Two panels 6 x 6 x 0.125 inches 2024-T3 nonclad were identified as 2M5SICIT2+1Y plus one panel 0.5 x 6 x 0.25 inches was identified as "S72" and 2 panels 6 x 6 x 0.125 inches same alloy were identified as 2M5SICIT2+2Y and one small panel same alloy was identified as "S73".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data recorded during Sealing & Anodizing (4/24/75)

(1) Concentration of $K_2Cr_2O_7$ (C1)	2.504% by wt.
(2) Type of seal water used (S1)	D1
(3) Temperature of seal solution (T2)	204°F
(4) Time in seal solution (t1)	8 minutes
(5) Time in seal solution (t2)	17 minutes
(6) pH of seal solution	3.98
(7) Temperature of Anodize Solution	95.5°F
(8) Total Amps. DC	6.8
(9) Amps. per square foot	2.71
(10) Total surface area	361.2 in. ²

37. Two panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5SICIT2+1Y plus one panel same alloy 0.5 x 6 x 0.125 inch was identified as "S74" and two panels 6 x 6 x 0.125 inch of the same alloy were identified as 7M5SICIT2+2Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S75".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/24/75)

(1) Concentration of $K_2Cr_2O_7$ (C1)	2.504% by wt.
(2) Type of water used (S1)	D1
(3) Temperature of seal solution (T2)	205°F
(4) Time in the seal solution (t1)	8 minutes
(5) Time in the seal solution (t2)	17 minutes
(6) pH of seal solution	3.98
(7) Temperature of Anodizing Solution	95°F
(8) Total Amps. DC	7.0
(9) Amps. per square foot	2.79
(10) Total surface area	same

38. Two panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 2M5SIC2T2+1Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S76", and two panels same size and alloy were identified as 2M5SIC2T2+2Y, plus one panel same size and alloy were identified as "S77".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/24/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.83% by Wt.
(2) Type of water for seal (S1)	D1
(3) Temperature of seal solution (T2)	204°F
(4) Time in seal solution (t1)	8 minutes
(5) Time in seal solution (t2)	17 minutes
(6) pH of seal solution	4.03
(7) Temperature of Anodizing solution	95°F
(8) Total Amps. DC	6.8
(9) Total surface area	Same

39. Two panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 7M5SIC2T2+1Y plus one panel 0.5 x 6 x .125 inches as identified as "S78" and two panels same size and alloy were identified as 7M5SIC2T2+2Y plus one small panel same alloy was identified as "S79".

a. The process procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/24/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	5.83% by Wt.
(2) Type of water for seal solution (S1)	D1
(3) Temperature of seal solution (T2)	204°F
(4) Time in seal solution (t1)	8 minutes
(5) Time in seal solution (t2)	17 minutes
(6) pH of seal solution	4.03

(7) Temperature of Anodizing solution	96°F
(8) Total Amps. DC	7.0
(9) Total surface area	same

40. Two panels 6 x 6 x 0.125 inches of 2024-T3 nonclad were identified as 215SICIT1+1Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S80" and two panels same size and alloy were identified as 215SICIT1+2Y plus one panel 0.5 x 6 x .125 inches of the same alloy was identified as "S81".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing and Anodizing (4/24/75)

(1) Concentration of $K_2Cr_2O_7$ (C1)	2.504% by Wt.
(2) Type of water for seal (S1)	DI
(3) Temperature of seal solution (T1)	184°=186°F
(4) Time in seal solution (t1)	8 minutes
(5) Time in seal solution (t2)	17 minutes
(6) pH of seal solution	3.98
(7) Temperature of Anodizing solution	95°F

(Last 15 Min. 95°F)

(8) Total Amps. DC	6.7
(9) Total surface area	561.2 in ²

41. Two panels 6 x 6 x 0.125 Inches of 7075-T6 nonclad were identified as 715SICIT1+1Y plus one panel 0.5 x 6 x 0.125 inch same alloy were identified as "S82" and two panels same size and alloy were identified as 715SICIT1+2Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S83".

a. The processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Sealing & Anodizing (4/25/75)

(1) Concentration of $K_2Cr_2O_7$ (C1)	2.504% by Wt
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(2) Type of water for seal (S1)	D1
(3) Temperature of seal solution (T1)	185.5°F
(4) Time in seal solution (t1)	8 minutes
(5) Time in seal solution (t2)	17 minutes
(6) pH of seal solution	3.98
(7) Temperature of Anodizing solution	95.5°F-96°F
(8) Total Amps. DC	7.0
(9) Total surface area	same

42. Two panels 6 x 6 x 0.125 inches of nonclad 2024-T3 were identified as 215SIC2T1+1Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S84" and two panels 6 x 6 x 0.125 inches of the same alloy were identified as 215SIC2T1+2Y plus one panel same alloy 0.5 x 6 x 0.125 inches was identified as "S85".

a. Data Recorded During Sealing and Anodizing (4/25/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.30% by Wt.
(2) Type of water for seal solution (S1)	D1
(3) Temperature of seal solution (T1)	184°F
(4) Time in seal solution (t1)	8 minutes
(5) Time in seal solution (t2)	17 minutes
(6) pH of seal solution	4.03
(7) Temperature of Anodizing solution	96°F
(8) Total Amps. DC	6.7
(9) Total surface area	same

43. Two panels 6 x 6 x 0.125 inches of 7075-T6 nonclad were identified as 705SIC2T1+1Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S86" and two panels same size and alloy were identified as 705SIC2T1+2Y plus one panel 0.5 x 6 x 0.125 inches of the same alloy was identified as "S87".

a. Data Recorded During Sealing & Anodizing (4/25/75)

(1) Concentration of $K_2Cr_2O_7$ (C2)	6.305% by Wt.
(2) Type of water for seal (S1)	DI
(3) Temperature of seal (T1)	182°F
(4) Time in seal solution (+1)	8 minutes
(5) Time in seal solution (+2)	17 minutes
(6) pH of seal solution	4.03
(7) Temperature of Anodizing solution	95°F
(8) Total Amps. DC	6.9
(9) Total surface area	same

APPENDIX "G"

Chromic Acid Anodizing Process Parameter Confirmation

1. Three panels, 6 x 6 x 0.125 inches of 2024-T3 nonclad alloy, were identified as 2M6CIVIT1+1, and one panel, 0.5 x 6 x 0.125 inches of the same alloy, was identified as "S87".

NOTE: All panels will be 6 x 6 x 0.125 inches nonclad—two for bonding and one for salt spray test. All small panels will be 0.5 x 6 x 0.125 inches, which will be used for instrumental analysis.

Panel Identification Code

2 = 2024-T3 alloy	7 = 7075-T6 alloy
M = MDC CrO ₃ process	6 = Task 106
C1 = 6 oz/gal free CrO ₃	C2 = 8 oz/gal free CrO ₃
V1 = 17 volts DC	V2 = 15 volts DC
T1 = 85°F	T1 = 100°F
t1 = 25 minutes	t2 = 40 minutes

a. Processing Procedure

- (1) The same as for Appendix F.1.a.(1)-(8).
- (2) The panels were anodized as indicated by identification code numbers.
- (3) The median range of the sealing parameter as shown below was used for all sealing processes.
 - (a) $K_2Cr_2O_7$ concentration = 5% by weight
 - (b) Temperature of seal = 190°F
 - (c) Time in seal solution = 10 minutes
 - (d) pH of seal solution = as obtained in makeup
 - (e) Type of water used = DI

b. Data Recorded During Anodizing & Sealing (5/2/75)

(1) Free CrO_3 (CI)	5.74 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodizing solution (TI)	83°F
(4) Time anodized (tI)	25 minutes
(5) Total surface area	254.6 in ²
(6) Total amps DC	2.7
(7) Amps per square foot	1.53
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(9) Temperature of seal solution	189°F
(10) Appearance of anodic film	iridescence

2. Three panels of 7075-T6 were identified as 7M6CIVIT1t1, and one small panel was identified as "S89".

a. Air treating procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/2/75)

(1) Free CrO_3 (CI)	5.74 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodizing solution (TI)	84°F
(4) Time anodized (tI)	25 minutes
(5) Total surface area	same
(6) Total amps DC	3
(7) Amps per square foot	1.70
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(9) Temperature of seal solution	193°-198°F
(10) Appearance of anodic film	iridescence

3. Two panels of 2024-T3 were identified as 2M6CIVIT1t2, and one small panel was identified as "S88".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/2/75)

(1) Free CrO_3 (C1)	5.74 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solution (T1)	84.5°F
(4) Time anodized (t2)	40 minutes
(5) Total surface area	167 in ²
(6) Total amps DC	2.3
(7) Amps per square foot	1.98
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(9) Temperature of seal solution	190°F
(10) Appearance of anodic film	Iridescence

4. Two panels of 2024-T3 were identified as 2M6CIV2T1t1, and one small panel was identified as "S91".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/2/75)

(1) Free CrO_3 (C1)	5.74 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T1)	84.5°F
(4) Time anodized (t1)	25 minutes
(5) Total surface area	same
(6) Total amps DC	2.3
(7) Amps per square foot	same
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(9) Temperature of seal solution	190°F

5. Two panels of 2024-T3 were identified as 2M6CIV2T1t2, and one small panel was identified as "S93".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/2/75)

(1) Free CrO_3 (C1)	5.74 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T1)	85°F
(4) Time anodized (t2)	40 minutes
(5) Total surface area	same
(6) Total amps DC	2.2
(7) Amps per square foot	1.70
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(9) Temperature of seal solution	190°F

b. Two panels of 7075-T6 were identified as 7M6CIV1T1t2, and one small panel was identified as "S96".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/5/75)

(1) Free CrO_3 (C1)	5.74 oz/gal
(2) Volts DC (V1)	17 minutes
(3) Temperature of anodizing solution (T1)	87°F
(4) Time anodized (t2)	25 minutes
(5) Total surface area	167 in^2
(6) Total amps DC	2.2
(7) Amps per square foot	1.90
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(9) Temperature of seal solution	192°F

7. Two panels of 7075-T6 were identified as 7M6CIV2T1t1, and one small panel was identified as "S92".

3. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/5/75)

(1) Free CrO_3 (CI)	5.74 oz/gal
(2) Volts DC (VI)	23
(3) Temperature of anodizing solution (TI)	86°F
(4) Time anodized (t2)	25 minutes
(5) Total amps DC	2.4
(6) Total surface area	same
(7) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(8) Temperature of seal solution	189°F

8. Two panels of 7075-T6 were identified as 7M6CIV2T1t2, and one small panel was identified as "S94".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/5/75)

(1) Free CrO_3 (CI)	5.74 oz/gal
(2) Volts (VI)	23
(3) Temperature of anodizing solution (TI)	85.5°F
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	2.4
(6) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.98% by weight
(7) Temperature of seal solution	190°F

9. Two panels of 2024-T3 were identified as 2M6CIVIT2t1, and one small panel was identified as "S97".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/6/75)

(1) Free CrO_3 (CI)	5.64 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodizing solution (T2)	99°F
(4) Time anodized (t1)	25 minutes

(5) Total amps DC	3.6
(6) Amps per square foot	3.10
(7) Concentration of $K_2Cr_2O_7$	5.007% by weight
(8) Temperature of seal solution	191°F
(9) Total surface area	167 in ²

10. Two panels of 2024-T3 were identified as 2M6CIVIT2t2, and one small panel was identified as "S98".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/6/75)

(1) Free CrO_3 (C1)	5.64 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solution (T2)	99.5°-100°F
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	3.7
(6) Concentration of $K_2Cr_2O_7$	5.007% by weight
(7) Temperature of seal solution	190°F

11. Two panels of 2024-T3 were identified as 2M6CIV2T2t1, and one small panel was identified as "S99".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/6/75)

(1) Free CrO_3 (C1)	5.64 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T2)	100°F
(4) Time anodized (t1)	25 minutes
(5) Total amps DC	4.0
(6) Concentration of $K_2Cr_2O_7$	5.007% by weight
(7) Temperature of seal solution	191°F

12. Two panels of 2024-T3 were identified as 2M6CIV2T2t2, and one small panel was identified as "S100".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/6/75)

(1) Free CrO_3 (C1)	5.64 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T2)	100°F
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	6.2
(6) Amps per square foot	3.58
(7) Total surface area	249.5 in ²
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	5.007% by weight
(9) Temperature of seal solution	191°F

13. Two panels of 7075-T6 were identified as 7M6CIV1T2t1, and one small panel was identified as "S101".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/7/75)

(1) Free CrO_3 (C1)	5.68 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solution (T2)	100°F
(4) Time anodized (t1)	25 minutes
(5) Total amps DC	3.7
(6) Amps per square foot	3.19
(7) Total surface area	167 in ²
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	5.007% by weight
(9) Temperature of seal solution	189°F

14. Two panels of 7075-T6 were identified as 7M6CIV1T2+2, and one small panel was identified as "S102".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/7/75)

(1) Free CrO_3 (C1)	5.68 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solution (T2)	99.5°F
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	3.7
(6) Total surface area	
(7) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	5.997% by weight
(8) Temperature of seal solution	190°F

15. Two panels of 7075-T6 were identified as 7M6CIV2T2+1, and one small panel was identified as "S103".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/7/75)

(1) Free CrO_3 (C1)	5.68 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T2)	100°F
(4) Time anodized (t1)	25 minutes
(5) Total amps DC	4.4
(6) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	5.997% by weight
(7) Temperature of seal solution	190°F

16. Two panels of 7075-T6 were identified as 7M6CIV2T2+2, and one small panel was identified as "S104".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/7/75)

(1) Free CrO_3 (C1)	5.68 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T2)	100°F
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	6.6
(6) Amps per square foot	3.81
(7) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	5.007% by weight
(8) Temperature of seal solution	189°F
(9) Total surface area	249.5 in^2

17. Three panels 2024-T3 were identified as 2M6C2VIT1t1; one of the three to be used for salt spray test. One small panel was identified as "S105".

NOTE: The low temperature (T1) was changed to 90F to determine the cause for this iridescence coating.

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/8/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solution (T1)	89.5°F
(4) Time anodized (t1)	25 minutes
(5) Total surface area	249.5 in^2
(6) Total amps DC	4.0
(7) Amps per square foot	2.31
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.81% by weight
(9) Temperature of seal solution	190°F
(10) Appearance	iridescence

18. Two panels of 2024-T3 were identified as 2M6C2V1T1t2, and one small panel was identified as "SI06".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/8/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodized solution (T1)	90°F
(4) Time anodized (t2)	40 minutes
(5) Total surface area	167 in^2
(6) Total amps DC	2.8
(7) Amps per square foot	same
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.81% by weight
(9) Temperature of seal solution	190°F
(10) Appearance	iridescence

19. Two panels of 2024-T3 were identified as 2M6C2V2T1t1, and one small panel was identified as "SI07".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/8/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T1)	90°F
(4) Time anodized (t1)	25 minutes
(5) Total amps DC	2.9
(6) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.81% by weight
(7) Temperature of seal solution	190°F
(8) Appearance of anodic film	slight iridescence

20. Two panels of 2024-T3 were identified as 2M6C2V2T1t2, and one small panel was identified as "SI08".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/8/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T1)	89.5°
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	3.0
(6) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.819 by weight
(7) Temperature of seal solution	190°F

21. Three panels of 7075-T6 were identified as 7M6C2V1T1t1, and one small panel was identified as "SI09".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/9/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solution (T1)	89°-90.5°F
(4) Time anodized (t1)	25 minutes
(5) Total surface area	249.5 in^2
(6) Total amps	4.2
(7) Amps per square foot	2.42
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.819 by weight
(9) Temperature of seal solution	190°F

22. Two panels of 7075-T6 were identified as 7M6C2V1T1t2, and one small panel was identified as "SI10".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/9/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V1)	17
(3) Temperature of anodizing solutions (T1)	90°F
(4) Time anodized (t2)	40 minutes
(5) Total amps DC	3.0
(6) Total surface area	167 in ²
(7) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.81% by weight
(8) Temperature of seal solution	190°F

23. Two panels of 7075-T6 were identified as 7M6C2V2T1t1, and one small panel was identified as "S11".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/9/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T1)	92°F
(4) Time anodized (t1)	25 minutes
(5) Total amps	3.3
(6) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.81% by weight
(7) Temperature of seal solution	190°F

24. Two panels of 7075-T6 were identified as 7M6C2V2T1t2, and one small panel was identified as "S112".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/9/75)

(1) Free CrO_3 (C2)	8.02 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T1)	91°F
(4) Time anodized (t2)	40 minutes

- | | |
|-----------------------------------|-----------------|
| (5) Total amps DC | 3.3 |
| (6) Concentration of $K_2Cr_2O_7$ | 4.81% by weight |
| (7) Temperature of seal solution | 189°F |

25. Two panels of 2024-T3 were identified as 2M6C2VIT2+1, and one small panel was identified as "S113"

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/12/75)

- | | |
|--|-------------------------|
| (1) Free CrO_3 (C2) | 7.98 oz/gal |
| (2) Volts DC (V1) | 17 |
| (3) Temperature of anodizing solution (T2) | 100°F |
| (4) Time anodized (+1) | 25 minutes |
| (5) Total surface area | 167.8 in ² |
| (6) Total amps DC | 4.0 |
| (7) Amps per square foot | 3.43 |
| (8) Concentration of $K_2Cr_2O_7$ | 4.81% by weight |
| (9) Temperature of seal solution | 192°F |
| (10) Appearance of anodic film | very little iridescence |

26. Two panels of 2024-T3 were identified as 2M6C2VIT2+2, and one small panel was identified as "S114".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/12/75)

- | | |
|--|-------------|
| (1) Free CrO_3 (C2) | 7.98 oz/gal |
| (2) Volts DC (V1) | 17 |
| (3) Temperature of anodizing solution (T2) | 100°F |
| (4) Time anodized (+2) | 40 minutes |
| (5) Total surface area | same |
| (6) Total amps DC | 4.2 |

(7) Amps per square foot	same
(8) Concentration of $K_2Cr_2O_7$	4.81% by weight
(9) Temperature of seal solution	190°F
(10) Appearance of anodic film	no iridescence

27. Two panels of 2024-T3 were identified as 2M6C2V2T2+1, and one small panel was identified as "S115".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/12/75)

(1) Free CrO_3 (C2)	7.98 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodize solution (T2)	100°F
(4) Time anodized (t1)	25 minutes
(5) Total surface area	same
(6) Amps per square foot	same
(7) Total amps DC	4.6
(8) Concentration of $K_2Cr_2O_7$	4.81% by weight
(9) Temperature of seal solution	193°F
(10) Appearance of anodic film	no iridescence

28. Three panels of 2024-T3 were identified as 2M6C2V2T2+2—one panel for salt spray test—and one small panel was identified as "S116".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/12/75)

(1) Free CrO_3 (C2)	7.98 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodize solution (T2)	100°F
(4) Time anodized (t2)	40 minutes
(5) Total surface area	254 in ²

(6) Total amps DC	6.8
(7) Amps per square foot	3.86
(8) Concentration of $K_2Cr_2O_7$	4.81% by weight
(9) Temperature of seal solution	193°F

29. Two panels of 7075-T6 were identified as 7M6Q2VIT2+1, and one small panel was identified as "S117".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/13/75)

(1) Free CrO_3 (C2)	7.98 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodizing solution (T2)	99.5°F
(4) Time anodized (+1)	25 minutes
(5) Total surface area	167 in ²
(6) Total amps DC	4.2
(7) Amps per square foot	3.62
(8) Concentration of $K_2Cr_2O_7$	4.995% by weight
(9) Temperature of seal solution	193°F

30. Two panels of 7075-T6 were identified as 7M62VIT2+2, and one small panel was identified as "S118".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/13/75)

(1) Free CrO_3 (C2)	7.98 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodize solution (T2)	100°F
(4) Time anodized (+2)	40 minutes
(5) Total amps DC	4.2
(6) Total surface area	same

- | | |
|-----------------------------------|------------------|
| (7) Amps per square foot | same |
| (8) Concentration of $K_2Cr_2O_7$ | 4.995% by weight |
| (9) Temperature of seal solution | 190°F |

31. Two panels of 7075-T6 were identified as 7M6C2V2T2t1, and one small panel was identified as "S119".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/13/75)

- | | |
|--|------------------|
| (1) Free CrO_3 (C2) | 7.98 oz/gal |
| (2) Volts DC (V2) | 23 |
| (3) Temperature of anodize solution (T2) | 100°F |
| (4) Time anodized (t1) | 25 minutes |
| (5) Total amps DC | 4.5 |
| (6) Total surface area | same |
| (7) Amps per square foot | same |
| (8) Concentration of $K_2Cr_2O_7$ | 4.995% by weight |
| (9) Temperature of seal solution | 192°F |

32. Three panels of 7075-T6 were identified as 7M6C2V2T2t2—one was used for salt spray test—and one small panel was identified as "S120".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/13/75)

- | | |
|--|---------------------|
| (1) Free CrO_3 (C2) | 7.98 oz/gal |
| (2) Volts DC (V2) | 23 |
| (3) Temperature of anodize solution (T2) | 99.5°F |
| (4) Time anodized (t2) | 40 minutes |
| (5) Total amps DC | 7.3 |
| (6) Total surface area | 254 in ² |
| (7) Amps per square foot | same |
| (8) Concentration of $K_2Cr_2O_7$ | 4.995% by weight |
| (9) Temperature of seal solution | 192°F |

Extension of MDC Chromic Acid Anodizing Confirmation Matrix

As most favorable results were obtained with the processing conditions in 4th quadrant of the anodizing matrix, it was desired to extend the quadrant by increasing the temperature to 105°F as (T2) in order to determine if better results can be obtained.

1. Two panels of 2024-T3 was identified as 2M6C2VIT2+IX, and one small panel of the same alloy was identified as "SI24".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/21/75)

(1) Free CrO_3 (C2)	7.72 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodizing solution (T2)	104°-104.5°F
(4) Time anodized (t1)	25 minutes
(5) Total surface area	167 in ²
(6) Total amps DC	4.8
(7) Amps per square foot	4.14
(8) Concentration of $\text{K}_2\text{Cr}_2\text{O}_7$	4.995% by weight
(9) Temperature of seal solution	193°F
(10) Appearance of anodic film	looked like powder after rubbing; left no residue

2. Two panels of 2024-T3 was identified as 2M6C2VIT2+2X, and one small panel was identified as "SI25".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/21/75)

(1) Free CrO_3 (C2)	7.72 oz/gal
(2) Volts DC (VI)	17
(3) Temperature of anodizing solution (T2)	105°F

(4) Time anodized (t2)	40 minutes
(5) Total surface area	same
(6) Total amps DC	4.9
(7) Amps per square foot	same
(8) Concentration of $K_2Cr_2O_7$	4.995% by weight
(9) Temperature of seal solution	192°F
(10) Appearance of anodic film	same as before

3. Two panels of 2024-T3 were identified as 2M6C2V2T1X, and one small panel was identified as "S126".

a. Processing procedure was the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/21/75)

(1) Free CrO_3 (C2)	7.72 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T2)	104.5°-105°F
(4) Time anodized (t1)	25 minutes
(5) Total surface area	same
(6) Total amps DC	5.5
(7) Amps per square foot	4.74
(8) Concentration of $K_2Cr_2O_7$	4.995% by weight
(9) Temperature of seal solution	193°F
(10) Appearance of anodic film	same

4. Three panels of 2024-T3 were identified as 2M6C2V2T2+2X—one for salt spray test—and one small panel was identified as "S127".

a. Processing procedure is the same as described in Appendix F.1.a.

b. Data Recorded During Anodizing & Sealing (5/21/75)

(1) Free CrO_3 (C2)	7.72 oz/gal
(2) Volts DC (V2)	23
(3) Temperature of anodizing solution (T2)	105.5°F

(4) Time anodized (t ₂)	40 minutes
(5) Total surface area	same
(6) Total amps DC	8.4
(7) Amps per square foot	same
(8) Concentration of K ₂ Cr ₂ O ₇	4.995% by weight
(9) Temperature of seal solution	192°F
(10) Appearance of anodic film	same

**PABST
FULL SCALE
DEVELOPMENT COMPONENT**

FINISH SPECIFICATION F-527

TYPE FINISH SPECIFICATION

F-527

PABST

FULL SCALE

DEVELOPMENT COMPONENT

DATE OF ISSUE

NEW: MARCH 9, 1977

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PAGE REVISION LETTER RECORD

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MATERIAL LIST

<u>Identifying No.</u>	<u>Description 1/</u>	<u>Vendor</u>	<u>Vendor Code No.</u>
DMS 1786	FR Primer	DeSoto, Inc.	515-700
DMS 2115	Aliphatic Polyurethane Coating, Color 17875	DeSoto, Inc.	821X330
DMS 2169, Type 1	Metal Bond Primer	American Cyanamid	BR127
MIL-P-23377	Epoxy- Polyamide Primer	DeSoto, Inc.	513-703
MIL-S-A1733	Sealant	Product Research & Chemical Corp.	PR1431G and PR1436G

Footnote:

1/ Color number is from Federal Standard FED-STD-595.

APPLICABLE DOUGLAS PROCESS STANDARDS (DPS's)

DPS 1.950, SPEO P-001	Metal Adhesive Bonding
DPS 2.50	Sealing Methods
DPS 3.317	Preservation & Protection of Machined Precision Parts
DPS 3.320	Protection of Sheet Metal (Raw Stock and Parts)
DPS 3.320-1	Protection of Milled Skins
DPS 4.50-36	Epoxy (FR) Coating System
DPS 4.50-138	Epoxy Primer, DMS 2104
DPS 4.50-165	Aliphatic Polyurethane Coating DMS 2115
DPS 9.45	Conversion Coatings for Aluminum
DPS 11.01	Chromic Acid Anodizing Aluminum
DPS 11.08, SPEO P-001	Phosphoric Acid Anodizing of Aluminum

SECTION 1.0

INTRODUCTION

1.0 INTRODUCTION

1.1 PURPOSE

This document specifies the finishes and their respective processes that are to be employed in the fabrication and assembly of the PABST Full Scale Development Component.

1.2 SCOPE

This specification shall be interpreted as establishing the requirements necessary for fastener installation with sealant together with the coating and sealing of details and assemblies. The Development Component will be involved in fatigue testing without adverse environment.

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SECTION 2.0
FUSELAGE FINISHES

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2.0 FUSELAGE FINISHES

The PABST fuselage (Full Scale Development Component) is defined as, and to include, the structural and nonstructural detail parts and assemblies just aft of the cockpit section and through the center section of the airframe.

2.1 GENERAL FUSELAGE FINISH REQUIREMENTS

Protect all raw stock and details per DPS 3.317, 3.320 and 3.320-1 as required. The finish requirements are applicable to detail parts after all fabrication operations have been completed.

2.1.1 MECHANICALLY ATTACHED ALUMINUM DETAIL PARTS

2.1.1.1 Nonclad 2000 and 7000 Series Aluminum Details

(A)

Chromic acid anodize MIL-A-8625, Type I - DPS 11.01 (except Frames J197732 and J197733 and Floor Planks J118631 - apply MIL-C-5541, C.C. Coating - DPS 9.45)

Apply MIL-P-23377 epoxy polyamide primer - DPS 4.50-138

2.1.1.2 All Clad Alloys and Nonclad 6000 Series Aluminum Details

Apply MIL-C-5541, C.C. or M.C. Coating - DPS 9.45

Apply MIL-P-23377 epoxy polyamide primer - DPS 4.50-138 or
apply DMS 1786 FR primer - DPS 4.50-36

2.1.2 METAL ADHESIVE BONDED ASSEMBLIES

2.1.2.1 Aluminum Detail Parts

Phosphoric acid anodize - DPS 11.08, SPEO P-001

Apply DMS 2169, Type I metal bond primer - DPS 1.950, SPEO P-001

2.1.2.2 Assembly After Bonding

2.1.2.2.1 General

Apply MIL-P-23377 epoxy polyamide primer - DPS 4.50-138
(all surfaces that will be mechanically joined and faying
surface sealed)

2.1.2.2.2 Part Number J197714 Assembly

Apply MIL-P-23377 epoxy polyamide primer to all surfaces -
DPS 4.50-138

After mechanical assembly, apply white polyurethane topcoat,
DMS 2115-17875 per DPS 4.50-165 to all surfaces except
panel splice joints

2.1 GENERAL FUSELAGE FINISH REQUIREMENTS (Cont'd)**2.1.3 ASSEMBLY SEALING REQUIREMENTS****2.1.3.1 Detail Parts or Assemblies Mechanically Attached with Fasteners that Penetrate the Exterior Skin**

Faying surface seal with MIL-S-81733 sealant (PR1431G) - DPS 2.50

2.1.3.2 All Fasteners that Penetrate a Metal Adhesive Bond Line or Exterior Skin

Wet Install with MIL-S-81733 sealant (PR1431G or PR1436G) -
DPS 2.50

PROCESS ENGINEERING ORDER				SHEET 1 OF 1		F-527				
DOUGLAS AIRCRAFT COMPANY LONG BEACH, CALIFORNIA ROBERT H. DOUGLAS PRESIDENT				DATE	1	DOCUMENT REVISED				
				DATE	2	ADVANCE PEO				
				DATE	3	SERIAL PEO				
				DATE 3-9-77	4	NEW/REVISED RELEASE	NEW			
				DATE	5	REISSUE TO REVISE				
TITLE PABST FULL SCALE DEVELOPMENT COMPONENT				M & E <i>J. R. Hollinger</i>		PEO MADE BY MF				
HANDLING INSTRUCTIONS (HH): 2 COMPLY AT SPECIFIED EFFECTIVITY 3 COMPLY WHEN INVOKED BY DRAWING OR OTHER AUTHORITY 4 COMPLY AS NEEDED 5 NOTED				ROUTING CODE M	RELEASE					
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All	PABST	3	Full Scale Development							
			Component							

EFFECTIVE ON WORK PERFORMED AT OR FOR THE DOUGLAS AIRCRAFT COMPANY

This PEO is authority to release new Finish Specification F-527.

PROCESS ENGINEERING ORDER DOUGLAS AIRCRAFT COMPANY <small>1000 BOEING BLVD. TORRANCE, CALIF. 90503</small> ROSCOE H. DOUGLAS <small>DOUGLAS AIRCRAFT COMPANY</small>				SHEET 1 OF 1		F-527	
				DATE	1	DOCUMENT REVISED	
				DATE 8-24-77	2	ADVANCE PED	A
				DATE	3	SERIAL PED	
				DATE	4	NEW REVISED RELEASE	
				DATE	5	REISSUE TO REVISE	
TITLE				M. J. J. J.		PRO MADE BY: M. J. J. J.	
PABST FULL SCALE DEVELOPMENT COMPONENT						J. R. Hollinger	
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				SCHEDULE	PROGRAM ENGINE	DRAFTING MANUAL TO BE REVISED	
				DOG		YES NO	
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MODEL				Not Required			
HI NO				ACTION COPIES			
EFFECTIVITY				STANDARD DISTRIBUTION LIST			
2.1.1.1	PABST	3	Full Scale Development	As Required			
			Component				

EFFECTIVE ON WORK PERFORMED AT OR FOR THE DOUGLAS AIRCRAFT COMPANY

This Advance PED "A" is authority to change Finish Specification F-527 (NEW), dated 5-9-77.

Changes pages

1, 2, 3, 4, 5, 6, 7, 8

Changes paragraph

2.1.1.1

Reason:

Add Floor Planks P/N J118651 to the exception from chromic acid anodizing nonclad 2000 and 2000 series aluminum details.

Reason:

Size of subject parts prohibits their being chromic acid anodized.

PABST FINISH SPECIFICATION

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MATERIALS LIST

<u>Identifying No.</u>	<u>Description</u>	<u>Vendor</u>	<u>Vendor Code No.</u>
DMS 2165, Type I	Metal Bond Primer	American Cyanamid	BR127
MIL-P-23377	Epoxy- Polyamide Primer	DeSoto, Inc.	513-703
MIL-C-83266 (#17875) DN9374	Gloss White Urethane Topcoat	DeSoto, Inc.	821X330
MIL-S-81733	Sealant	Product Research & Chemical Corp.	PRI436G

APPLICABLE DOUGLAS PROCESS STANDARDS (DPS's)

DPS 1.950	Metal Adhesive Bonding
DPS 2.50	Sealing Methods
DPS 4.50-138	Epoxy Primer, DMS 2104
DPS 4.50-165	Aliphatic Polyurethane Coating, DMS 2115
DPS 9.45	Conversion Coating for Aluminum
DPS 11.01	Chromic Acid Anodize
DPS 11.08	Phosphoric Acid Anodize

SECTION 1.0
INTRODUCTION

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1.0 INTRODUCTION

1.1 PURPOSE

This document specifies the finishes and their respective processes that are to be employed in the fabrication and assembly of the PABST fuselage section.

1.2 SCOPE

This specification shall be interpreted as establishing the minimum requirements necessary to assure adequate protection from corrosion.

SECTION 2.C
FUSELAGE FINISHES

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2.0 FUSELAGE FINISHES

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2.1.3.3 Exterior Butt Gaps

2.1.3.4 Metal Adhesive Bonded Trimmed Edges

2.1.4 ASSEMBLY ORGANIC COATING REQUIREMENTS

2.1.4.1 Nonmold Line Surfaces

2.1.4.2 Exterior Mold Line Surfaces

2.0 FUSELAGE FINISHES

The PABST fuselage is defined as, and to include, the structural and nonstructural detail parts and assemblies just aft of the nose section and through the center section of the airframe.

2.1 GENERAL FUSELAGE FINISH REQUIREMENTS

The finish requirements are applicable to detail parts after all fabrication operations have been completed.

2.1.1 MECHANICALLY ATTACHED ALUMINUM DETAIL PARTS

2.1.1.1 Nonclad 2000 and 7000 Series Aluminum Details

MIL-A-8625, Type I - DPS 11.01

MIL-P-23377 epoxy polyamide primer - DPS 4.50-138

2.1.1.2 All Clad Alloys and Nonclad 6000 Series Aluminum Details

MIL-C-5541, M.C. Coating - DPS 9.45

MIL-P-23377 epoxy polyamide primer - DPS 4.50-138

2.1.2 METAL ADHESIVE BONDED ASSEMBLIES

2.1.2.1 Aluminum Detail Parts

Phosphoric acid anodize - DPS 11.00

DMS 2169, Type I metal bond primer - DPS 1.050

2.1.2.2 Assembly After Bonding

MIL-P-23377 epoxy polyamide primer - DPS 4.50-138

(all surfaces except exterior mold line)

2.1.3 ASSEMBLY SEALING REQUIREMENTS

2.1.3.1 Mechanically Attached Detail Parts

Faying surface seal with MIL-S-81733 sealant - DPS 2.50

2.1.3.2 All Fasteners that Penetrate a Metal Adhesive Bond

Net Install with MIL-S-81733 sealant - DPS 2.50

2.1.3.3 Exterior Butt Gaps

MIL-S-81733 sealant - DPS 2.50

2.1 GENERAL FUSELAGE FINISH REQUIREMENTS (Cont'd)

2.1.3 ASSEMBLY SEALING REQUIREMENTS (Cont'd)

2.1.3.4 Metal Adhesive Bonded Trimmed Edges

MIL-C-5541, M.C. Coating - DPS 9.45

Fillet seal with MIL-S-81733 sealant - DPS 2.50

2.1.4 ASSEMBLY ORGANIC COATING REQUIREMENTS

2.1.4.1 Nonmold Line Surfaces

After assembly and sealing, apply MIL-P-23377 epoxy polyamide primer per DPS 4.50-138. In addition, apply MIL-C-83286 DN9374 white polyurethane topcoat per DPS 4.50-165 to surfaces below the main cabin floor.

2.1.4.2 Exterior Mold Line Surfaces

After assembly and sealing, apply MIL-P-23377 epoxy polyamide primer per DPS 4.50-138 and MIL-C-83286 DN9374 white polyurethane topcoat per DPS 4.50-165.